Haley & Aldrich, Inc.
5755 Granger Road
Suite 320
Cleveland, OH 44131-1442

Tel: 216.739.0555
Fax: 216.739.0560
HaleyAldrich.com

17 December 2011
File No. 37294-001

Hazardous Waste Program, Superfund Section
Missouri Department of Natural Resources
1738 East Elm St.
Jefferson City, MO 65101

Attention: Dennis Stinson, Chief

Subject: Site Investigation Workplan
Former Leeds Assembly Plant - Northern Parcel
South of 6817 Stadium Drive
Kansas City, Missouri

Dear Mr Stinson:

On behalf of Motors Liquidation Corporation, please find the Site Investigation Workplan (Workplan) for the Former Leeds Assembly Plant - Northern Parcel. The Workplan details the proposed initial stage of investigation activities intended to determine the nature and extent of potential impact at or from the Site and addressing their effects on human health and the environment. The Workplan also provides the supporting documents for the investigation and potential future evaluation activities.

We would like to meet with you at your convenience to review the Workplan. Please contact Mr. David M. Favero, P.G. at 217-522-6714 or dfavero@ameritech.net to schedule a meeting date.

If you have any questions or require additional information, please contact us.

Sincerely yours,
HALEY & ALDRICH, INC.

Daniel R. Putz
Project Manager

David J. Hagen
Vice President

Enclosure – Site Investigation Workplan

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SITE INVESTIGATION WORKPLAN
FORMER LEEDS ASSEMBLY PLANT - NORTHERN PARCEL
MOTORS LIQUIDATION COMPANY
KANSAS CITY, MISSOURI

by

Haley & Aldrich, Inc.
Cleveland, Ohio

for

Motors Liquidation Company
Detroit, Michigan

File No. 37294-001
17 December 2011

HALEY & ALDRICH

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APPENDIX B - Quality Assurance Project Plan
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3.	Proposed Location Plan
4.	Proposed Location Plan with January 2000 Geophysical Survey Anomalies
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1. INTRODUCTION

On behalf of Motors Liquidation Company (MLC), the following presents the Site Investigation Workplan (Workplan) for the Former Leeds Assembly Plant - Northern Parcel (MLC ID 1007) located south of 6817 Stadium Drive in Kansas City, Missouri (Site) (Figure 1). The Site is owned by MLC. The purpose of this Workplan is to propose soil and groundwater testing locations and methods to characterize the nature and extent of impacts from the historical filling operations on the Site.

1.1 Site Setting

The approximate 15-acre Site is bordered to the North by the Leeds Industrial Park (the former General Motors Leeds assembly facility, not owned by MLC), to the east by a Kansas City Southern railroad, to the south by MLC's Former Leeds Assembly Plant - Southern Parcel (owned by MLC, ID 1109), and to the west by a Missouri Pacific railroad. A portion of the Site's southwest corner is bordered by the Blue River. The Site is zoned M2 Heavy Industrial and generally lies within an industrial area; however, a mixed commercial and residential area lies to the northwest of the Site beyond a railroad. The Site is in Jackson County.

1.2 Site History

The Site was used for farming until the Leeds Assembly plant was constructed on the parcel adjacent to the north of the Site in 1928. Around that time railroads were constructed across the Site. The railroad siding shed near the eastern edge of the property boundary was built between 1957 and 1965. The former propand storage area in the northwest corner of the site was built between 1969 and 1978. Fill placement on the east half and northern edge of the Site occurred between 1950 and 1952. Fill placement in the southwestern portion of the Site occurred between 1952 and 1960. By 1963 nearly the entire Site was paved and has remained largely unchanged since.

1.3 Geologic Setting

The Site is located in the Disected Till Plains physiographic province, which was formed by glacial scouring and deposition by the Laurentide ice sheet during the Pre-Illinoian Stage glaciation and later accumualtion of loess during the Wisconsin Stage.

The Site is located on the edge of the alluvial valley of the Blue River and is covered nearly entirely with asphalt pavement. Fill material composed of anthropomorphic material mixed with native material lies below the pavement. Fill thickness ranges from a few feet to over 15 feet thick. Native soil underlying the fill consists of fine grained alluvial materials such as silty clay, clayey silt and fine sand. A thin (1 to 5 feet) deposit of gravely sandy clay is present at the base of the overburden deposit. Overburden thickness is approximately 50 to 60 feet. Shale bedrock of the Pleasanton Group has been encountered beneath the overburden.

1.4 Hydrogeologic Setting

The Site is generally flat with an elevation of approximately 761 feet along the northern, eastern and southern property boundaries and approximately 758 or 759 in the central portion of the Site and along the western property boundary. The piezometric surface has been found at approximately 15 feet below grade in both wells screened across the water table and in wells screened at the bottom of the overburden. Groundwater flow direction has been shown to be west toward the Blue River. All monitoring wells which could be located at the time were abandoned in 1994 or 1996.

1.5 Site Investigation History

In 1990 and 1991 four monitoring wells and two borings were installed on the Site along the southern property boundary. These were part of an investigation into a former waste disposal area on the parcel south of the Site. Results of this study can be found in the Report on the Site Characterization of the Former Waste Disposal Area[1]. The wells were abandoned in 1994 and the details of the abandonment can be found in the Well Abandonment Report[2].

Buried drums were discovered at the Site in 1999 during trenching activities for utilities installation (Figure 2). Later that year 13 soil borings were drilled around the area of the drum discovery and the report Subsurface Investigation Report, Buried Drum Discovery[3], concluded that buried drums were limited to a 45 foot by 45 foot by 12 foot deep area.

Based on the findings of the soil boring program, an excavation was planned to remove drums. The excavation was started in December 1999 but the extent of drums was not delineated. Trenches started from the drum discovery area delineated the extent of drums to the west, but did not define the extent to the north, south and east before going beyond the intended scope of the project. The trenching was halted in the delineation phase. Samples were collected from a buried drum containing paint, a green material, and a red layer of material. Four soil samples were collected by GM and MDNR from the bottom (approximately 15 feet deep) of the main excavation (the starting point of the trenching). A sample of a greenish material was collected from a drum recovered from the trench south of the main excavation. At the conclusion of the trenching, four samples were collected from the suspected clean soil stockpile. After filling and repaving of the trenches a geophysical survey was performed in the vicinity of the drum discovery. The findings of the trenching investigation and geophysical survey can be found in the Remedial Action Report, Recovery of Buried Drums[4].

In 2003 an investigation of historical documents and aerial photographs was performed to try and determine the nature and extent of historic fill operations on the Site. The report concluded that filling operations on a large portion of the eastern portion the Site occurred from 1950 to 1952 and filling operations on a large portion of the western portion the Site occurred from 1952 to 1960. Results of this investigation can be found in the Report of Historical Activities in the Waste Disposal Site Vicinity[5].

1.6 Current and Anticipated Future Site Use

The Site is currently zoned M2a – Heavy Industrial and is used for vehicle and equipment storage. It is anticipated that the Site will continue to be used for industrial purposes.

2. INVESTIGATION ACTIVITIES

The proposed investigation will consist of a soil investigation and a groundwater investigation. The investigation has been designed to determine the nature and extent of the fill material and impacts from the fill material. The proposed investigation takes into account the findings of past investigations and is focused of areas identified in the past as known or suspect fill areas.

2.1 Soil Investigation

The soil investigation will be completed by installing 22 test pits in the locations specified on Figure 3. The test pit locations are also shown on Figure 4 with the January 2000 Geophysical Survey Anomalies. The total depth of the test pits will be approximately 1 to 3 feet into native soil unless the capability of the excavotor is exceeded by depth or obstructions. One to three soil samples will be collected from each test pit to be determined by field conditions such as: total depth, visual observations, and photoionization detector readings. The spoils from the test pits will be collected on polyethelene sheeting and the open excavation and spoils will be photographed. The spoils will be returned to the excavation, compacted by the excavator, and the pavement will be replaced. The soil samples will be sent to the analytical laboratory for analysis of VOCs, SVOCs, and the RCRA 8 Metals.

All sample collection, sample handling and sample preservation will be performed in accordance with the Field Sampling Plan (Appendix A) and the Quality Assurance Project Plan (Appendix B).

2.2 Groundwater Investigation

The groundwater investigation will be completed by installing 22 shallow (up to 20 feet deep) and 2 deep wells (up to 35 feet deep) in the locations specified on Figure 3. The monitoring well locations are also shown on Figure 4 with the January 2000 Geophysical Survey Anomalies. The monitoring wells will be installed in two rounds as indicated on Figures 3 and 4. The first round of wells will be installed, developed and sampled before installing the second round of wells to confirm if the locations are appropriate. The monitoring wells will be developed and sampled using low-flow methods for VOCs, SVOCs, and the RCRA 8 Metals.

All sample collection, sample handling and sample preservation will be performed in accordance with the Field Sampling Plan (Appendix A) and the Quality Assurance Project Plan (Appendix B).

2.3 Health and Safety

A Site specific Health and Safety plan has been prepared which includes Site engineering controls, monitoring requirements, action levels, personal protection equipment requirements, a list of emergency contacts, and a map to the nearest health care facility. The Health and Safety Plan is included in Appendix C.

2.4 Schedule

A Site specific schedule is included as Figure 5.

REFERENCES

1. Burns & McDonnell; Report on the Site Characterization of the Former Waste Disposal Site, Leeds Assembly Plant, General Motors Corporation, Kansas City, Missouri; January 1992.

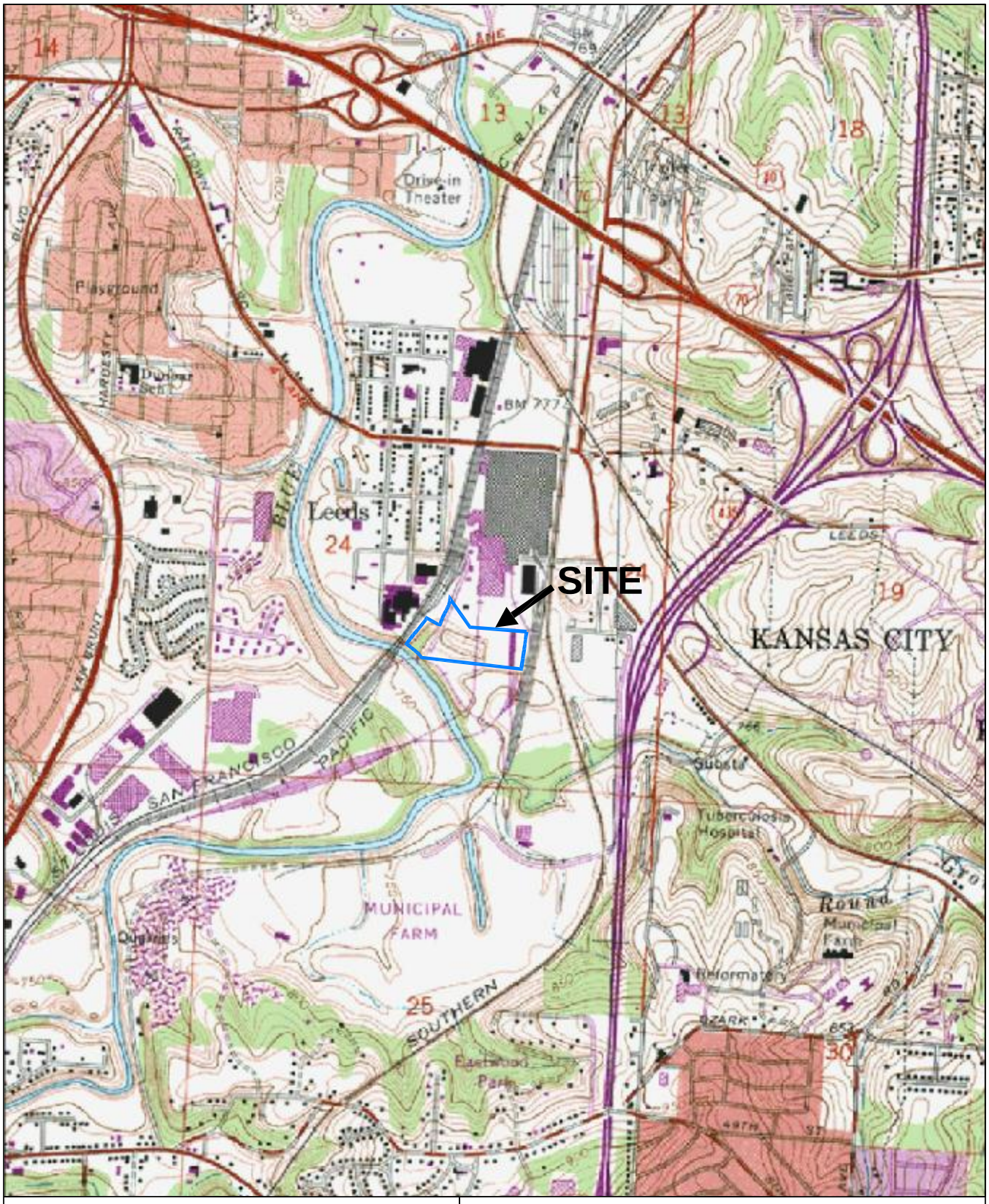
2. Burns & McDonnell; Former General Motors Leeds Plant, Kansas City, Missouri, Well Abandonment Report; September 1997.

3. Burns & McDonnell; Subsurface Investigation Report, Buried Drum Discovery, Former General Motors Leeds Assembly Plant, Kansas City, Missouri; July 1999.

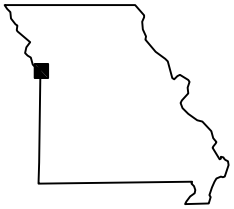
4. Burns & McDonnell; Remedial Action Report, Recovery of Buried Drums, Former General Motors Leeds Assembly Plant, Kansas City, Missouri; August 2000.

5. Burns & McDonnell; Report of Historical Activities in the Waste Disposal Site Vicinity, Former GM Leeds Assembly Plant, Kansas City, Missouri; October 2003.

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SITE COORDINATES: 39°3'9"N 94°30'19"W



U.S.G.S. QUADRANGLE: KANSAS CITY, MO

HALEY & ALDRICH

MOTORS LIQUIDATION COMPANY
FORMER LEED'S ASSEMBLY PLANT-NORTHERN PARCEL
SOUTH OF 6817 STADIUM DRIVE
KANSAS CITY, MISSOURI

PROJECT LOCUS

SCALE: 1:24000
NOVEMBER 2010

FIGURE 1

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DRUM DISCOVERY AREA

DRAINAGE DITCH

TRAIN BRIDGE

BLUE RIVER

SITE

RAIL LOADING BUILDING

LEGEND:

- PROPERTY LINE
- RAIL ROAD TRACKS
- FORMER RAIL ROAD TRACKS
- RIVER / SWALE
- FENCE
- EXISTING STRUCTURE

NOTES:

1. ALL LOCATIONS APPROXIMATE.
2. THIS FIGURE WAS GENERATED WITH THE USE OF SEVERAL SOURCES:
   - MAIN SITE FEATURES WERE DIGITIZED FROM AN AERIAL IMAGE OBTAINED FROM GOOGLE EARTH PRO.
   - PROPERTY LINES WERE DIGITIZED FROM MAPS OBTAINED FROM JACKSON COUNTY AUDITOR'S WEBSITE.
   - EXPLORATION LOCATIONS, AND DRUM AREA WERE DIGITIZED FROM MAPS PROVIDED BY THE CLIENT.

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APPROXIMATE SCALE IN FEET

HALEY & ALDRICH

MOTORS LIQUIDATION COMPANY
FORMER LEED'S ASSEMBLY PLANT-NORTHERN PARCEL
SOUTH OF 6817 STADIUM DRIVE
KANSAS CITY, MISSOURI

SITE PLAN

SCALE: AS SHOWN
SEPTEMBER 2010

FIGURE 2

G:\37294 - MLC LEEDS\GLOBAL\CAD\DRAWINGS\37294-SITEPLAN_R1.DWG

DEEP

DRUM DISCOVERY AREA

DEEP

DRAINAGE DITCH

SITE

RAIL LOADING BUILDING

DEEP

BLUE RIVER

TRAIN BRIDGE

LEGEND:

- PROPERTY LINE
- RAIL ROAD TRACKS
- FORMER RAIL ROAD TRACKS
- RIVER / SWALE
- FENCE
- EXISTING STRUCTURE
- ABANDONED MONITORING WELL LOCATION
- PROPOSED (FIRST ROUND) WATER TABLE MONITORING WELL LOCATION
- DEEP PROPOSED (SECOND ROUND) DEEP MONITORING WELL LOCATION
- PROPOSED (SECOND ROUND) MONITORING WELL LOCATION
- PROPOSED TEST PIT LOCATION

NOTES:

1. ALL LOCATIONS APPROXIMATE.
2. THIS FIGURE WAS GENERATED WITH THE USE OF SEVERAL SOURCES:
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   - EXPLORATION LOCATIONS, AND DRUM AREA WERE DIGITIZED FROM MAPS PROVIDED BY THE CLIENT.

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APPROXIMATE SCALE IN FEET

HALEY & ALDRICH

MOTORS LIQUIDATION COMPANY
FORMER LEED'S ASSEMBLY PLANT-NORTHERN PARCEL
SOUTH OF 6817 STADIUM DRIVE
KANSAS CITY, MISSOURI

PROPOSED LOCATION PLAN

SCALE: AS SHOWN
DECEMBER 2010

FIGURE 3

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DRUM DISCOVERY AREA

DRAINAGE DITCH

TRAIN BRIDGE

BLUE RIVER

SITE

RAIL LOADING BUILDING

DEEP

LEGEND:

- PROPERTY LINE
- RAIL ROAD TRACKS
- FORMER RAIL ROAD TRACKS
- RIVER / SWALE
- FENCE
- EXISTING STRUCTURE
- ABANDONED MONITORING WELL LOCATION
- PROPOSED (FIRST ROUND) WATER TABLE MONITORING WELL LOCATION
- DEEP PROPOSED (SECOND ROUND) DEEP MONITORING WELL LOCATION
- PROPOSED (SECOND ROUND) MONITORING WELL LOCATION
- PROPOSED TEST PIT LOCATION

NOTES:

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   - EXPLORATION LOCATIONS, AND DRUM AREA WERE DIGITIZED FROM MAPS PROVIDED BY THE CLIENT.

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APPROXIMATE SCALE IN FEET

HALEY & ALDRICH

MOTORS LIQUIDATION COMPANY
FORMER LEED'S ASSEMBLY PLANT-NORTHERN PARCEL
SOUTH OF 6817 STADIUM DRIVE
KANSAS CITY, MISSOURI

PROPOSED LOCATION PLAN WITH JANUARY 2000 GEOPHYSICAL SURVEY ANOMALIES

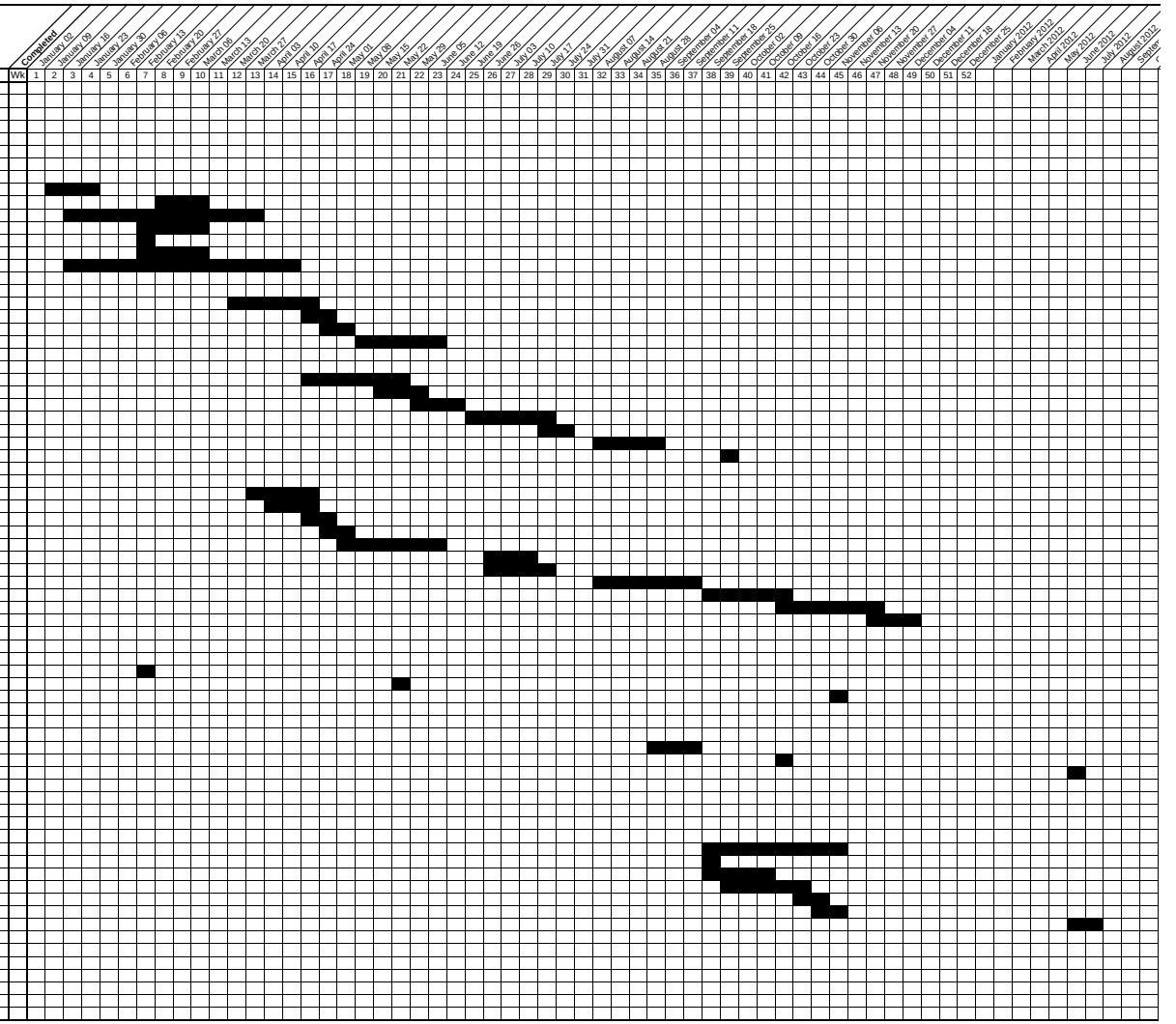
SCALE: AS SHOWN
OCTOBER 2010

FIGURE 4

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Figure 5
2011 PROJECT SCHEDULE AND MILESTONES
MLC#1007 Leed's Northern Parcel

Task ID	Project Tasks and Milestones	Ownership	Dependent Task(s)	Dates Start	Dates Finish	Duration
1	**Site Investigation Work Plan**					
1.01	Draft Field Investigation Work Plan	D. Putz		08/02/10 Mon	10/01/10 Fri	61
1.02	MLC Review	D. Favero		10/01/10 Fri	11/15/10 Mon	46
1.03	Finalize and Submit Field Investigation Work Plan	D. Putz		11/15/10 Mon	11/22/10 Mon	8
1.04	MoDNR Review and Comment	MoDNR	1.01	11/22/10 Mon	12/23/10 Thu	32
1.05	Revise/Finalize Field Investigation Work Plan	D. Putz	1.02	12/23/10 Thu	12/31/10 Fri	9
2	**Site Investigation**					
2.01	Field Investigation - Initial	D. Putz	1.03	01/10/11 Mon	01/28/11 Fri	19
2.02	Field Investigation - Follow-up	D. Putz	6.02	02/21/11 Mon	03/11/11 Fri	19
2.03	Laboratory Analysis	D. Putz	2.01/2.02	01/17/11 Mon	04/01/11 Fri	75
2.04	Groundwater Sampling	D.Putz		02/14/11 Mon	03/11/11 Fri	26
2.04.01	Sampling	D.Putz		02/14/11 Mon	02/18/11 Fri	5
2.04.02	Lab Analysis	D.Putz		02/16/11 Wed	03/11/11 Fri	24
2.05	Data Evaluation	D. Putz	2.02	01/17/11 Mon	04/15/11 Fri	89
3	**Site Investigation Report**					
3.01	Field Investigation Data Report	D. Putz	2.03	03/21/11 Mon	04/22/11 Fri	33
3.02	MLC Review	D. Favero	3.01	04/22/11 Fri	04/29/11 Fri	8
3.03	Revise/Finalize Field Investigation Data Report	D. Putz	3.02	04/29/11 Fri	05/06/11 Fri	8
3.04	MoDNR Review and Comment	MoDNR	3.03	05/09/11 Mon	06/08/11 Wed	31
4	**Deed Restriciton**					
4.01	Identify Areas for Restriction	D. Putz	2.03	04/22/11 Fri	05/23/11 Mon	32
4.02	Develop Draft Deed Restriction	D. Putz	4.01	05/16/11 Mon	05/30/11 Mon	15
4.03	MLC and Legal Review of Draft Deed Restriction	D. Favero	4.02	05/30/11 Mon	06/13/11 Mon	15
4.04	MoDNR Deed Restriciton Review	MoDNR	4.03	06/20/11 Mon	07/18/11 Mon	29
4.05	Revise Deed Restriction	D.Putz	4.04	07/18/11 Mon	07/25/11 Mon	8
4.06	Survey Restriction Areas	D. Putz	4.05	08/08/11 Mon	08/29/11 Mon	22
4.07	File Deed Restriction	D.Putz	4.06	09/26/11 Mon	09/26/11 Mon	1
5	**Soil and Drum Removal**					
5.01	Identify Areas for Remediation	D.Putz	2.03	03/28/11 Mon	04/18/11 Mon	22
5.02	Develop Remedial Action Work Plan	D.Putz	5.01	04/04/11 Mon	04/22/11 Fri	19
5.03	MLC Review	D. Favero	5.02	04/22/11 Fri	04/29/11 Fri	8
5.04	Finalize and Submit Remedial Action Work Plan	D.Putz	5.03	04/29/11 Fri	05/06/11 Fri	8
5.05	MoDNR Review and Comment	MoDNR	5.04	05/06/11 Fri	06/07/11 Tue	33
5.06	Revised Remedial Action Work Plan	D.Putz	5.05	06/27/11 Mon	07/11/11 Mon	15
5.07	Contractor Bidding and Selection	D.Putz	5.06	06/27/11 Mon	07/18/11 Mon	22
5.08	Implement Soil and Drum Removal Activities	D.Putz	5.07	08/08/11 Mon	09/16/11 Fri	40
5.09	Prepare Remedial Action Report	D.Putz	5.08	09/19/11 Mon	10/21/11 Fri	33
5.10	MoDNR Review and Comment	MoDNR	5.09	10/21/11 Fri	11/22/11 Tue	33
5.11	Revise/Finalize Remedial Action Report	D.Putz	5.08	11/22/11 Tue	12/06/11 Tue	15
6	**Project Management, Meetings with MoDNR**					
6.01	Initial Project Meeting	D. Favero	1.01	11/03/10 Wed	11/03/10 Wed	
6.02	Review of Initial Field Results Meeting/Call	D. Favero	2.01	02/16/11 Wed	02/16/11 Wed	
6.02	Field Investigation Result and Remedial Action Meeting	D. Favero	2.03	05/25/11 Wed	05/25/11 Wed	
6.04	Remedial Action Follow-up Meeting	D. Favero	5.07	11/08/11 Tue	11/08/11 Tue	
7	**Cap Monitoring & Maintenance**					
7.01	2010 Initial Cap Inspection	D.Putz		10/25/10 Mon	10/27/10 Wed	3
7.02	2011 Cap Sealing	D.Putz		09/02/11 Fri	09/16/11 Fri	15
7.03	2011 Cap Inspection	D.Putz		10/17/11 Mon	10/19/11 Wed	3
7.04	2012 Cap Inspection	D.Putz		06/11/12 Mon	06/11/12 Mon	1
7.05	2013 Cap Inspection	D.Putz		06/10/13 Mon	06/12/13 Wed	3
7.06	2014 Cap Inspection	D.Putz		06/09/14 Mon	06/09/14 Mon	1
7.07	2015 Cap Inspection	D.Putz		06/08/15 Mon	06/08/15 Mon	1
8	**Groundwater Monitoring and Reporting**					
8.01	2011 Q3 Groundwater Sampling and Reporting	D. Putz		09/19/11 Mon	11/11/11 Fri	54
8.01.01	Sampling	D.Putz		09/19/11 Mon	09/23/11 Fri	5
8.01.02	Lab Analysis	D.Putz		09/21/11 Wed	10/14/11 Fri	24
8.01.03	Draft Groundwater Summary Report	D.Putz		09/26/11 Mon	10/28/11 Fri	33
8.01.04	MLC Review	D. Favero		10/28/11 Fri	11/04/11 Fri	8
8.01.05	Finalize and Submit Groundwater Report	D. Putz		11/04/11 Fri	11/11/11 Fri	8
8.04	2012 Annual Groundwater Sampling and Reporting	D.Putz		06/11/12 Mon	07/27/12 Fri	47
8.05	2013 Annual Groundwater Sampling and Reporting	D.Putz		06/10/13 Mon	07/26/13 Fri	47
8.06	2014 Annual Groundwater Sampling and Reporting	D.Putz		06/09/14 Mon	07/25/14 Fri	47
8.07	2015 Annual Groundwater Sampling and Reporting	D.Putz		06/08/15 Mon	07/24/15 Fri	47
9	**Agency Oversight and Fees**	D. Favero				



Notes:
1. 30-day review cycle is assumed from MoDNR review and comment and no MoDNR approval required in Brownfields/Voluntary Cleanup Program (BVCP).

Figure 5
2011 PROJECT SCHEDULE AND MILESTONES
MLC#1007 Leed's Northern Parcel

Task ID	Project Tasks and Milestones	Ownership	Dependent Task(s)	Dates		
				Start	Finish	Duration
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1.03	Finalize and Submit Field Investigation Work Plan	D. Putz		11/15/10 Mon	11/22/10 Mon	8
1.04	MoDNR Review and Comment	MoDNR	1.01	11/22/10 Mon	12/23/10 Thu	32
1.05	Revise/Finalize Field Investigation Work Plan	D. Putz	1.02	12/23/10 Thu	12/31/10 Fri	9
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2.03	Laboratory Analysis	D. Putz	2.01/2.02	01/17/11 Mon	04/01/11 Fri	75
2.04	Groundwater Sampling	D.Putz		02/14/11 Mon	03/11/11 Fri	26
2.04.01	Sampling	D.Putz		02/14/11 Mon	02/18/11 Fri	5
2.04.02	Lab Analysis	D.Putz		02/16/11 Wed	03/11/11 Fri	24
2.05	Data Evaluation	D. Putz	2.02	01/17/11 Mon	04/15/11 Fri	89
3	**Site Investigation Report**					
3.01	Field Investigation Data Report	D. Putz	2.03	03/21/11 Mon	04/22/11 Fri	33
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4	**Deed Restriciton**					
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4.03	MLC and Legal Review of Draft Deed Restriction	D. Favero	4.02	05/30/11 Mon	06/13/11 Mon	15
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4.06	Survey Restriction Areas	D. Putz	4.05	08/08/11 Mon	08/29/11 Mon	22
4.07	File Deed Restriction	D.Putz	4.06	09/26/11 Mon	09/26/11 Mon	1
5	**Soil and Drum Removal**					
5.01	Identify Areas for Remediation	D.Putz	2.03	03/28/11 Mon	04/18/11 Mon	22
5.02	Develop Remedial Action Work Plan	D.Putz	5.01	04/04/11 Mon	04/22/11 Fri	19
5.03	MLC Review	D. Favero	5.02	04/22/11 Fri	04/29/11 Fri	8
5.04	Finalize and Submit Remedial Action Work Plan	D.Putz	5.03	04/29/11 Fri	05/06/11 Fri	8
5.05	MoDNR Review and Comment	MoDNR	5.04	05/06/11 Fri	06/07/11 Tue	33
5.06	Revised Remedial Action Work Plan	D.Putz	5.05	06/27/11 Mon	07/11/11 Mon	15
5.07	Contractor Bidding and Selection	D.Putz	5.06	06/27/11 Mon	07/18/11 Mon	22
5.08	Implement Soil and Drum Removal Activities	D.Putz	5.07	08/08/11 Mon	09/16/11 Fri	40
5.09	Prepare Remedial Action Report	D.Putz	5.08	09/19/11 Mon	10/21/11 Fri	33
5.10	MoDNR Review and Comment	MoDNR	5.09	10/21/11 Fri	11/22/11 Tue	33
5.11	Revise/Finalize Remedial Action Report	D.Putz	5.08	11/22/11 Tue	12/06/11 Tue	15
6	**Project Management, Meetings with MoDNR**					
6.01	Initial Project Meeting	D. Favero	1.01	11/03/10 Wed	11/03/10 Wed	
6.02	Review of Initial Field Results Meeting/Call	D. Favero	2.01	02/16/11 Wed	02/16/11 Wed	
6.02	Field Investigation Result and Remedial Action Meeting	D. Favero	2.03	05/25/11 Wed	05/25/11 Wed	
6.04	Remedial Action Follow-up Meeting	D. Favero	5.07	11/08/11 Tue	11/08/11 Tue	
7	**Cap Monitoring & Maintenance**					
7.01	2010 Initial Cap Inspection	D.Putz		10/25/10 Mon	10/27/10 Wed	3
7.02	2011 Cap Sealing	D.Putz		09/02/11 Fri	09/16/11 Fri	15
7.03	2011 Cap Inspection	D.Putz		10/17/11 Mon	10/19/11 Wed	3
7.04	2012 Cap Inspection	D.Putz		06/11/12 Mon	06/11/12 Mon	1
7.05	2013 Cap Inspection	D.Putz		06/10/13 Mon	06/12/13 Wed	3
7.06	2014 Cap Inspection	D.Putz		06/09/14 Mon	06/09/14 Mon	1
7.07	2015 Cap Inspection	D.Putz		06/08/15 Mon	06/08/15 Mon	1
8	**Groundwater Monitoring and Reporting**					
8.01	2011 Q3 Groundwater Sampling and Reporting	D. Putz		09/19/11 Mon	11/11/11 Fri	54
8.01.01	Sampling	D.Putz		09/19/11 Mon	09/23/11 Fri	5
8.01.02	Lab Analysis	D.Putz		09/21/11 Wed	10/14/11 Fri	24
8.01.03	Draft Groundwater Summary Report	D.Putz		09/26/11 Mon	10/28/11 Fri	33
8.01.04	MLC Review	D. Favero		10/28/11 Fri	11/04/11 Fri	8
8.01.05	Finalize and Submit Groundwater Report	D. Putz		11/04/11 Fri	11/11/11 Fri	8
8.04	2012 Annual Groundwater Sampling and Reporting	D.Putz		06/11/12 Mon	07/27/12 Fri	47
8.05	2013 Annual Groundwater Sampling and Reporting	D.Putz		06/10/13 Mon	07/26/13 Fri	47
8.06	2014 Annual Groundwater Sampling and Reporting	D.Putz		06/09/14 Mon	07/25/14 Fri	47
8.07	2015 Annual Groundwater Sampling and Reporting	D.Putz		06/08/15 Mon	07/24/15 Fri	47
9	**Agency Oversight and Fees**	D. Favero				

Notes:
1. 30-day review cycle is assumed from MoDNR review and comment and no MoDNR approval required in Brownfields/Voluntary Cleanup Progra

APPENDIX A

Field Sampling Plan

FIELD SAMPLING PLAN

FORMER LEEDS ASSEMBLY PLANT NORTHERN PARCEL
MOTORS LIQUIDATION COMPANY
KANSAS CITY, MISSOURI

by

Haley & Aldrich, Inc.
Cleveland, Ohio

for

Motors Liquidation Company
Detroit, Michigan

File No. 37294-001
October 22, 2010

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APPENDIX B - Encore Sampling Method

1. INTRODUCTION

This Field Sampling Plan (FSP) is submitted as an attachment to and forms part of the Investigation Work Plan submitted by Motors Liquidation Company for the Former Leeds Assembly Plant Northern Parcel (Facility). This Investigation Work Plan was prepared to detail the investigation work necessary to characterize potential releases of hazardous waste and hazardous constituents at or from the Site and assessing their effects on human health and the environment. The FSP was prepared to establish field procedures for field data collection at the Site. These standard operating procedures (SOPs) are intended to provide the procedures necessary to meet the objectives of the investigation.

2. FIELD PROGRAM

The field program has been designed to acquire the necessary data to meet the Data Quality Objectives (DQOs) outlined in the Quality Assurance Project Plan (QAPP). Haley & Aldrich will perform oversight and consulting during the field investigation. Data necessary to complete the investigation will be collected by: soil/sediment sampling, groundwater/surface water sampling, hydraulic conductivity testing, water level studies and additional sampling/analysis as needed.

These standard operating procedures (SOPs) may be modified or changed as required, dependent on site conditions, equipment limitations, or limitations imposed by the procedure. If the procedures employed differ from the SOP, the deviations will be documented.

The primary objective of sampling activities is to characterize environmental conditions accurately to evaluate their impact on human health and the environment. The sampling will be conducted so that samples collected will retain, as much as possible, their original physical form and chemical composition.

3. OPERATING PROCEDURES

The following Operating Procedures are included in Appendix A this Field Sampling Plan.

1.0 Initial Site Reconnaissance Surveys
- 1.1 Utility Clearance
- 1.2 Field Data Recording – Field Books, Log Forms, and Electronic Data

2.0 Subsurface Investigations
- 2.1 Drilling Techniques/Background Information
- 2.2 Soil Borings
- 2.3 Borehole Abandonment/Sealing
- 2.4 Soil Classification
- 2.5 Soil Vapor Monitoring Points

3.0 Monitoring Wells
- 3.1 Well Construction Materials
- 3.2 Procedures for Overburden Monitoring Well Installation
- 3.3 Well Decommissioning Procedures
- 3.4 Well Development Procedures

4.0 Aquifer Characterization
- 4.1 Manual Water Level Measurement Procedure
- 4.2 In-Situ Hydraulic Conductivity (Slug Testing) Procedure

5.0 Sample Collection for Laboratory Analysis
- 5.1 Soil Sample Collection for Laboratory Analysis
- 5.2 Groundwater Sample Collection for Laboratory Analysis
- 5.3 Non-Aqueous Phase Liquid (NAPL) Monitoring and Sample Collection
- 5.4 Surface Water Sampling
- 5.5 Sediment Sampling
- 5.6 Sample Handling and Shipping
- 5.7 Vertical Water Quality Profiling
- 5.8 Soil Vapor Sampling

6.0 Field Instruments – Use and Calibration

7.0 Equipment Decontamination

8.0 Waste Characterization – (Investigation-Derived Waste)

9.0 Sump Inspection

APPENDIX A

Standard Operating Procedures

1.0 INITIAL SITE RECONNAISSANCE SURVEYS

1.1 UTILITY CLEARANCE

INTRODUCTION

Invasive field investigation activities such as drilling, soil gas surveys, test excavation or remedial construction activities require location of underground utilities prior to initiating work. Such clearance is sound practice in that it minimizes the potential for damage to underground facilities and more importantly, is protective of the health and safety of personnel. Under no circumstances will invasive activities be allowed to proceed without obtaining proper utility clearance by the appropriate public agencies and/or private entities. **This clearance requirement applies to all work on both public and private property, whether located in a dense urban area or a seemingly out-of-the-way rural location.**

The responsibility of obtaining this clearance lies with the Consultant or Contractor performing the work.

In most states such utility clearance is required by law, and obtaining clearance includes contacting a public or private central clearance agency via a "one-call" telephone service and providing them with proposed exploration location information. This is discussed in more detail herein. It is important to note that public utility agencies may not, and usually don't have information regarding utility locations on private property. As such, utility clearance on Delphi plant property must be cleared using available site drawings, and written approval must be obtained from plant personnel with appropriate knowledge of existing utilities.

PROCEDURES REFERENCED

- 2.0 Subsurface Investigations

PROCEDURE

- Before marking any proposed exploration or underground construction locations, it is critical that all readily-available information on underground utilities and structures be obtained. This includes publicly-available information as well as information in the possession of private landowners. Any drawings obtained must be reviewed in detail for information pertaining to underground utilities.

- Using the information obtained, the site should be viewed in detail for physical evidence of buried lines or structures, including pavement cuts and patches,

variation in or lack of vegetation, variations in grading, etc. (Care must also be taken to avoid overhead utilities as well). Presence of surface elements of buried utilities should be documented, such as manholes, gas or water service valves, catch basins, monuments or other evidence.

- Overhead utility lines must be taken into account when choosing exploration and excavation locations. Most states require a minimum of 10 ft. of clearance between equipment and energized wires. Such separation requirements may also be voltage-based and may vary depending on state or municipality regulations.

  In evaluating clearance from overhead lines, the same restrictions may apply to "drops", or wires on a utility pole connecting overhead and underground lines.

- Using the information obtained and observations made, *proposed* exploration or construction locations should be marked in the field. Marking locations can be accomplished using spray paint on the ground, stakes, or other means**. All markings of proposed locations should be made in white**, in accordance with the generally-accepted universal color code for facilities identification (AWMA 4/99):

  - White: Proposed Excavation or Drilling location
  - Pink: Temporary Survey Markings
  - Red: Electrical Power Lines, Cables, Conduit and Lighting Cables
  - Yellow: Gas, Oil, Steam, Petroleum or Gaseous Materials
  - Orange: Communication, Alarm or Signal Lines, Cables or Conduits
  - Blue: Potable Water
  - Purple: Reclaimed Water, Irrigation and Slurry Lines
  - Green: Sewers and Drain Lines

- In order to effectively evaluate the proposed locations with these entities, detailed, accurate measurements between the proposed locations and existing surface features should be obtained. Such features can be buildings, street intersections, utility poles, guardrails, etc.

- Obtaining the utility clearance generally involves two entities:
  - The designated "One-Call" underground facilities protection organization for the area; and
  - The landowner.

  Both entities must be contacted and the proposed locations evaluated in light of information available for existing underground facilities. The detailed measurement information described above will be required by the "one call" agency. The owners of the applicable, participating underground utilities are obligated to mark their respective facilities at the site in the colors described

above. Utility stake-out activities will typically not commence for approximately 72 hours after the initial request is made.

- The public and private utility entities generally only mark the locations of their respective underground facilities within public rights-of-way. Determination of the locations of these facilities on private property will be the responsibility of the project Consultant or Contractor. If available information does not contain sufficient detail to locate underground facilities with a reasonable amount of confidence, alternate measures may be appropriate, as described below. In some cases, the memory of a long-time employee of a facility on private property may be the best or only source of information. It is incumbent on the Consultant or Contractor to exercise caution and use good judgement when faced with uncertainty.

- **Notes: It is important to note that not all utilities are participants in the "one-call" agency or process. As such, inquiries must be made with the "one-call" agency to determine which entities do not participate, so they can be contacted independently.**

  Most utility Stake-outs have a limited time period for which they remain valid, typically two to three weeks. It is critical that this time period be taken into account to prevent expiration of clearance prior to completion of the invasive activities, and the need to repeat the stake-out process.

- Care must be exercised to document receipt of notice from the involved agencies of the presence or absence of utilities in the vicinity of the proposed locations.

  Most agencies will generally provide a telephone or fax communication indicating the lack of facilities in the project area. **If contact is not made by all of the agencies identified by the "one-call" process, do not assume that such utilities are not present. Re-contact the "one-call" agency to determine the status.**

- For complicated sites with multiple proposed locations and multiple utilities, it is advisable to arrange an on-site meeting with utility representatives. This will minimize the potential for miscommunication amongst the involved parties.

- Completion of the utility stake out process is not a guarantee that underground facilities will not be encountered in excavations or boreholes; in fact, most "one-call" agencies and individual utilities do not offer guarantees, nor do they accept liability for damage that might occur. Accordingly, it is advisable that any invasive activities proceed with extreme caution in the upper four to five feet in the event the clearance has failed to identified an existing facility. This may necessitate hand-excavation or probing to confirm potential presence of shallow

utilities. If uncertainty exists for any given utility, extra activities can be initiated to solve utility clearance concerns. These options include:

- Hand digging, augering or probing to expose or reveal shallow utilities and confirm presence and location. In northern climates this may require advancing to below frost line, typically at least four feet.
- Screening the proposed work areas with utility locating devices, and/or hiring a utility locating service to perform this task. The private utility locating service is a growing industry that has formed a national organization. The National Utility Locate Contractors Association (NULCA) can be reached at 715-635-6004.

EQUIPMENT

- White Spray paint
- Wooden stakes, painted white or containing white flagging
- Color-code key
- Available drawings

REFERENCE

- American Public Works Association, April 1999, Uniform Color Code (http://www.apwa.net/)

1.2 FIELD DATA RECORDING – FIELD BOOKS, LOG FORMS AND ELECTRONIC DATA

INTRODUCTION

This procedure describes protocol for documenting standard investigation activities in the field. Field data serves as the cornerstone for an environmental project, not only for site characterization but also for additional phases of investigation or remedial design. Inaccurate or incomplete field data may create significant problems and additional project costs. In addition, recorded field data becomes a legal record of project work, and should be approached with that in mind. Producing legally defensible data includes proper and appropriate recording of field data as it is obtained in a manner that will preserve it for future use.

This procedure provides guidelines for accurate, thorough collection and preservation of written and electronic field data.

PROCEDURES REFERENCED

- 6.0 Field Instruments - Use And Calibration

PROCEDURE

Typical field data to be recorded generally includes, but is not limited to, the following:

- general field observations;
- numeric field measurements and instrument readings;
- quantity estimates;
- sample locations and corresponding sample numbers;
- relevant comments and details pertaining to the samples collected;
- documentation of activities, procedures and progress achieved;
- contractor pay item quantities;
- weather conditions;
- a listing of personnel involved in site-related activities;
- a log of conversations, site meetings and other communications; and
- field decisions made and pertinent information associated with the decisions.

Written Field Data

Written field data is generally recorded on one of two media: A standardized, pre-printed field log form, or a bound field log book. In general, use of a field log form is preferable as it prompts field personnel to make appropriate observations and record data in a standardized format. This promotes completeness and consistency from one person to the next.

In the absence of an appropriate pre-printed form, the data should be recorded in an organized and structured manner in a dedicated project field log book. Log books must be hard-cover, bound so that pages cannot be added or removed, and should be made from high-grade 50% rag paper with a water-resistant surface.

The following are guidelines for use of field log forms and log books:

1. Information must be factual and complete. Do not abbreviate.

2. All entries will be made in black indelible ink with a ballpoint pen and will be written legibly. Do not use "rollerball" or felt tip-style pens, since the water-soluble ink can run or smear in the presence of moisture.

3. All pages in a log book must be consecutively numbered. Field log forms should also be consecutively numbered.

4. Each day's work must start a new log book page.

5. At the end of each day, the current log book page must be *signed and dated* by the field personnel making the entries.

6. When using field log forms, they must also be *signed and dated*.

7. Make data entries immediately upon obtaining the data. Do not make temporary notes in other locations for later transfer to log forms or log books; this only increases the potential for error or loss of data.

8. Entry errors are to be crossed out with a single line, dated and initialed by the person making the correction.

9. Do not leave blanks on log forms, if no entry is applicable for a given data field, indicate so with "NA" or a dash ("--").

10. At the earliest practical time, photocopies of log forms and log book pages should be made and placed in the project file as a backup in the event the book or forms are lost are damaged.
11. Log books should be dedicated to one project only, i.e., do not record data from multiple projects in one log book.

Electronic Data

Electronic data recording is widely used in environmental investigation and remediation projects. In general, it involves electronic measurement of field information through the use of monitoring instruments, sensors, gauges, and equipment controls. The following is a list of guidelines for proper recording and management of electronic field data:

1. Field data management should follow requirements of a project-specific data management plan (DMP), if one exists.

2. Use only instruments that have been calibrated in accordance with manufacturer's recommendations.

3. Personnel properly trained and experienced in the use of the equipment and software should only perform usage of instruments, controls and computers for the purpose of obtaining field data.

4. Use only fully licensed software on PCs and laptops.

5. Loss of electronic files may mean loss of irreplaceable data. Every effort should be made to back up electronic files obtained in the field as soon as practical. A backup file placed on a disk and kept in a separate location from the original will minimize the potential for loss.

6. Electronic files, once transferred from field instruments or laptops to office computers, should be protected if possible to prevent unwanted or inadvertent manipulation or modification of data. Several levels of protection are usually available for spreadsheets, including making a file "read-only" or assigning a password to access the file.

7. Protect floppy disks from exposure to moisture, excessive heat or cold, magnetic fields, or other potentially damaging conditions.

8. Remote monitoring is often used to obtain stored electronic data from site environmental systems. A thorough discussion of this type of electronic field data recording is beyond the scope of this SOP. Such on-site systems are generally capable of storing a limited amount of data as a comma-delimited or spreadsheet file. Users must remotely access the monitoring equipment files via modem or other access, and download the data. In order to minimize the potential for loss of data, access and downloading of data should be performed frequently enough to insure the data storage capacity of the remote equipment is not exceeded.

EQUIPMENT

- 5” by 7” National 407 Field Book, with high-grade 50% rag paper with water-resistant surface, hard-cover, or equivalent;
- Appropriate field log forms;
- Indelible ball point pen (do not use “rollerball” or felt-tip style pens);
- Straight edge;
- Pocket calculator;
- Laptop computer (if required).

REFERENCES

2.0 SUBSURFACE INVESTIGATIONS

2.1 DRILLING TECHNIQUES/BACKGROUND INFORMATION

INTRODUCTION

This section will provide a brief description of common methods for conducting subsurface investigations. It should be noted that every drilling technology has its limitations.

PROCEDURES REFERENCED

- 2.2 Soil Borings
- 2.3 Bedrock Coring
- 2.5 Soil Classification
- 2.6 Bedrock Logging and Classification
- 3.2 Overburden Wells
- 3.3 Top of Bedrock Wells
- 3.4 Deep Bedrock Wells

DRILLING METHODS

It is important that the drilling method or methods used minimize disturbance of subsurface materials and not contaminate the subsurface and groundwater. The actual drilling method would be dependent upon site-specific geologic conditions. It is important to note that the drilling equipment selected be decontaminated before and between borehole locations to prevent cross contamination (see SOP 7.0). Where possible drilling methods that minimize waste generation (soil cuttings), and waste water generation (decon water), should be selected for Delphi investigation/remedial tasks.

In other settings it may be desirable to dictate drilling procedures that minimize turbidity/maximize the ability to achieve sediment-free groundwater. Generally, rotosonic techniques or rotary spun casing techniques achieve these objectives, or oversizing the borehole/sand pack may be considered, as well.

Rotosonic Drilling

This method consists of a combination of rotation with high frequency vibration to advance a core barrel to a desired depth. Once the vibration is stopped, the core barrel is retrieved, and the sample is vibrated or hydraulically extracted into plastic sleeves or sample trays. Monitoring wells shall be installed through the outer casing with minimal formation disturbance and mixing of formation materials. Rotosonic drilling generally requires less time than more traditional methods and minimizes soil mixing and soil disturbance (preferred for well locations where low turbidity is an important objective).

Continuous, relatively undisturbed samples can be obtained through virtually any formation. Conventional sampling tools can be employed as attachments (i.e., hydropunch, split spoon, shelby tube, etc.). No mud, air, water, or other circulating medium is required. The rotosonic method can drill easily through formations such as rock, sand, clay, or glacial till. The main limitation of this method is the availability of equipment, the large area required (i.e., drill units are quite large), and costs.

Direct Push (Geoprobe™)

Direct push refers to the sampler being "pushed" into the soil material without the use of drilling to remove the soil. This method relies on the amount of the drill weight combined with percussion for advancement of the tool string. Discrete soil samples are continuously obtained as well groundwater and vapor samples can also be collected utilizing this method. Subsurface investigations typically probe to depths of 30 feet or more, although depths will vary based on site-specific geology. This method is used extensively for initial Site screening activities to delineate vertical and horizontal plume presence and can significantly reduce investigative costs. This method is becoming more popular due to the limited cuttings that are produced during the sampling process and the sampling process speed. The use of the Geoprobe™ 6600 also allows for the installation of 2-inch diameter monitoring wells.

Rotary Method

This method consists of a drill rod attached to a drill bit (soils: tricone, drag; rock: button studded, diamond studded) that rotates and cuts through the soils and rock. The cuttings produced are forced to the surface between the borehole wall and the drill rod by drilling fluids that generally consist of water, drilling mud, or air. The drilling fluids not only force the cuttings to the surface but also keep the drilling bit cool. Using rotary methods for well installations can be difficult, as it usually requires several steps to complete the installation. First, the borehole is drilled; then temporarily cased; then the well is installed; and then the temporary casing is removed. In some cases, the borehole may remain open without installing a casing but this will only occur in limited instances (i.e., cohesive soils).

i) Water Rotary

When using water rotary, the potable water supply shall be analyzed for contaminants of concern. Water rotary is the preferred rotary method since the potable water is the only fluid introduced into the borehole during drilling. However, the use of water as a fluid is generally only successful when drilling in cohesive soils. The use of potable water (only) also reduces well development time, when compared to mud rotary.

ii) Air Rotary (typically used in rock)

When using air rotary, the air compressor must have an in-line oil filter system assembly to filter the oil mixed with the air coming from the compressor. This will help eliminate contaminant introduction into the formation. The oil filter system shall be regularly inspected. Air compressors not having an in-line oil filter system are not acceptable for air rotary drilling. A cyclone velocity dissipater or similar air containment system shall also be used to funnel the cuttings to one location rather than letting the cuttings blow uncontrolled out of the borehole. Air rotary may not be an acceptable method for well installation where certain contaminants are present in the formation. Alternatively, it may be necessary to provide treatment for the air being exhausted from the borehole during the installation process.

iii) Mud Rotary

Mud rotary is the least preferred rotary method because contamination can be introduced into the borehole from the constituents in the drilling mud (i.e., Ohio, Michigan). The drilling muds are generally non-toxic and do not introduce contaminants into the borehole, however, it is possible for mud to commonly infiltrate and affect water quality by sorbing metals and polar organic compounds. Chemical composition and priority pollutants analysis may be obtained from the manufacturer. Mud rotary shall utilize only potable water and pure (no additives) bentonite drilling muds. The viscosity of the drilling mud shall be kept as low as possible in order to expedite well development. Proper well development is essential to ensure the removal of all the drilling mud and to return the formation to its previously undisturbed state.

Hollow-Stem Auger

The hollow-stem continuous-flight auger (HSA) is among the most frequently used in the drilling of monitoring wells (overburden wells) or for placement of overburden casings for bedrock wells.

The primary advantages of hollow-stem augering are that:

- generally, no additional drilling fluids are introduced into the formation;
- representative geologic soil samples can be easily obtained using split-spoon samples in conjunction with the hollow-stem augers; and
- monitoring wells can be installed through the augers eliminating the need for temporary borehole casings.

Disadvantages of hollow-stem augering are:

- creates problems for select parameters;
- large volumes of cuttings are typically generated;

- decon is fairly time consuming/labor intensive; and
- relatively slow when compared to direct-push methods (soil sampling tasks).

Installing monitoring wells through hollow-stem augers is a relatively simple process although precautions need to be taken to ensure that the well is properly backfilled. This can be particularly problematic in cases where flowing sand is present.

Hollow-stem augers are available with inside diameters of 2.5, 3.25, 4.0, 4.25, 6.25, 8.25, and 10.25 inches. The most commonly used are 4.25 inches for 2-inch (5 cm) monitoring wells and 6.25 inches for 4-inch (10 cm) monitoring wells. Boreholes can usually be drilled with hollow-stem augers to depths up to 100 feet (30 m) in unconsolidated clays, silts, and sands. Removing augers in flowing sand conditions while installing monitoring wells may be difficult since the augers have to be removed without being rotated. A bottom plug or pilot bit assembly should be utilized to keep out soils and/or water that have a tendency to plug the bottom of the augers during drilling. If flowing sands are encountered, potable water (analyzed once for contaminants of concern) may be poured into the augers to equalize the pressure to keep the formation materials and water from coming up into the auger once the bottom plug is removed.

Dual-Wall Reverse Circulation Air Method of Drilling

This method consists of two concentric strings of drill pipe (an outer casing and a slightly smaller inner casing). The outer drill pipe is advanced using rotary drilling with a donut-shaped bit attached to the dual casing string cuts an area only the width of the two casings and annulus between. Compressed air is continually forced down the annulus between the inner casing carrying the drill cuttings and groundwater. At the surface, the inner casing is connected to a cyclone hopper where the drill cuttings and groundwater fall out the bottom of the hopper, and air is disbursed out the top. The dual wall provides a fully cased borehole in which to install a monitoring well. The only soil or groundwater materials exposed at any time are those at the drill bit. Therefore, the potential for carrying contamination from one stratum to another is minimal. Depth-specific groundwater samples can be collected during drilling; however, since the groundwater is aerated, analysis for volatile compounds may not be valid.

Well Points

In some limited cases, well points (sand points) are driven into place without the use of augers. This method provides no information on the geologic condition (other than the difficulty of driving which may be related to formation density). Well points are most often used simply to provide dewatering of a geologic unit prior to excavation in the area. Well points are also used in monitoring shallow hydrogeologic conditions such as in stream beds.

REFERENCE

Numerous publications are available describing current monitoring well design and construction procedures.

1. Driscoll, F.G., 1986. Groundwater and Wells, 2nd Edition. Johnson Division.
2. Freeze, R.A. and Cherry, J.A., 1979. Groundwater. Prentice Hall, Inc.
3. EPA/625/6-90/0166 (July 1991), Handbook Ground Water Volume II:Methodology
4. National Water Well Association, 1989. Handbook of Suggested Practices for the Design and Installation of Groundwater Monitoring Wells
5. Environmental Protection Agency (1986), RCRA Groundwater Monitoring Technical Enforcement Guidance Document, OSWER-9950.1

In addition, the following ASTM publications apply:

1. ASTM D5474 Guide for Selection of Data Elements for Ground-Water Investigations
2. ASTM D5787 Practice for Monitoring Well Protection
3. ASTM D5521 Guide for Development of Ground-Water Monitoring Wells in Granular Aquifers
4. ASTM D5978 Guide for Maintenance and Rehabilitation of Ground-Water Monitoring Wells
5. ASTM D5299 Guide for Decommissioning of Ground Water Wells, Vadose Zone Monitoring Devices, Boreholes and Other Devices for Environmental Activities
6. ASTM D5092 Standard Practice for Design and Installation of Ground Water Monitoring Wells in an Aquifer.

2.2 SOIL BORINGS

INTRODUCTION

The following presents a description of the methods generally employed for the installation of boreholes and the collection of subsurface soil samples. Boreholes are typically installed to define geologic conditions for hydrogeologic and geotechnical evaluation; to allow the installation of monitoring wells and piezometers; and to allow the collection of subsurface soil samples (generally above the water table) for chemical analysis.

Several manual methods are available for the collection of shallow subsurface soil samples (e.g., hand augers, post-hole augers, vibratory hammers). However, the most common methods to advance boreholes are rotosonic drilling techniques, hollow-stem augers (HSA), or the use of a direct-push equipment. SOP 2.1 Drilling Techniques/Background, provides insight into the advantages/disadvantages of these drilling methods.

PROCEDURES REFERENCED

- 1.1 Utility Clearance
- 2.1 Drilling Techniques/Background Information
- 2.5 Soil Classification
- 2.6 Bedrock Logging and Classification
- 5.1 Soil Sample Collection

BOREHOLE REQUIREMENTS

The following activities must be undertaken prior to installing a borehole.

i) Obtain a site plan and any previous stratigraphic logs. Determine the exact number and location of boreholes to be installed and the depths of samples for chemical analysis.

ii) Coordinate lab services including:
   - glassware/sample jars;
   - cooler;

- shipping details;
- start date; and
- expected duration.

iii) Establish borehole locations in field using available landmark or by surveying methods if necessary.

iv) Arrange for utility clearance of franchised utilities and site utilities.

v) Determine notification needs with the Project Manager. Notify the regulatory groups, landowner, Delphi Facility personnel, and laboratory of the sampling event.

vi) Determine the methods for handling and disposal of drill cuttings, wash waters, and spent decontamination fluids.

Once the prior planning and preparation activities are completed, the borehole installation and subsurface soil sampling program can proceed. The typical work sequence is as follows:

- locating and marking of borehole locations (if not already completed);
- equipment decontamination;
- final visual examination of proposed drilling area for utility conflicts/final hand auger or post-hole check to verify utility absence;
- advancement of borehole and collection of the soil sample;
- field screening of soil sample;
- description of soil sample (Form 2.6-01 will be used to record data);
- sample preparation and packaging;
- abandonment of boreholes;
- surveying of borehole locations and elevations; and
- field note completion and review.

i) <u>Location and Marking of Boreholes/Final Visual Check</u>

The proposed borehole locations marked on the site plan are located in the field and staked. On most sites, this will likely be done several days in advance of the drill rig arriving on site. Unless boreholes are to be installed on a fixed grid, the proposed locations are usually placed at biased locations.

Once the final location for the proposed boring has been selected and utility clearances are complete, one last visual check of the immediate area should be performed before drilling proceeds. This last visual check should confirm the locations of any adjacent utilities (subsurface or overhead) and verification of adequate clearance. If gravity sewers or conduits exist in the area, any access manholes or chambers should be opened and the conduit/sewer alignments confirmed. Do not enter manholes unless confined space procedures are followed.

ii) Borehole Advancement

If possible, it is prudent to use a hand auger or post-hole digging equipment to a sufficient depth to verify the absence of buried utilities and pipelines. This procedure should clear the area to the full diameter of the drilling equipment that will follow. (**Only if underground utilities are suspected in the area, or requested by client.**)

If it is necessary to relocate any proposed borehole due to terrain, utilities, access, etc., the Field Coordinator/Task Manager must be notified and an alternate location will be selected. If moved, details of new location should be recorded in field log book.

Prior to use and between each borehole location at an environmental site, the drilling and sampling equipment must be decontaminated. All decontamination must be conducted in accordance with the project-specific plans or the methods presented in SOP 7.0

The clean augers/tooling are covered with clean plastic sheeting to prevent contact with foreign materials. For geotechnical, geologic, or hydrogeologic studies where contaminants will not be present, it is sufficient to clean the drilling equipment simply by removing the excess soils.

- Collection of soil samples is one of the most important considerations in selecting drilling methods. Therefore, the need for reviewing drilling techniques (SOP 2.1) and the Site objectives must first be considered. Soil Classification will be completed in accordance with SOP 2.5. Sections iii) and iv) describe borehole soil sampling procedures using direct-push tooling and hollow stem augering/split spoon sampling (Standard Penetration Testing - SPT), respectively.

iii) Direct-Push/Macro-Core™ Soil Sampler

The operation of the direct-push/Macro-Core™ Soil Sampler (or equivalent) consists of "pushing" the sampler into the subsurface and then retrieved using a direct-push soil probing machine. The collected soil core is contained within an internal soil liner (acetate, polyethylene or Teflon) and removed from the sampler once returned to the ground surface. Sampler length is variable depending on equipment available (2 ft., 4 ft., 5 ft.). Once the soil liner has been removed and the outer sampler de-coned, a new liner is inserted and the sampler reassembled. The clean sampler is then driven back down the same hole to collect the next soil sample.

The Macro-Core™ sampler can be used in either the open-tube or closed-point sampling mode. The open-core sample mode is most commonly used in stable soil conditions. In unstable soils, the piston rod point system prevents collapsed soil from entering the sampler as it is advanced back down the hole. Once at the sample depth, the piston rod is unthreaded and released. The sampler is then driven into the subsurface to fill the sampler with soil, the piston point rides on top of the soil, as it enters the sampler.

Once recovered the soil liner with collected soils is opened (cut lengthwise) and examined to collected soil screening information, soil logging information, and soils for chemical analysis.

iv) Standard Penetration Testing (SPT) Sampling and Testing Procedure

This method is used to obtain representative samples of subsurface soil materials and to determine a measure of the in situ relative density of the subsurface soils. The test methods described below must be followed to obtain accurate SPT values. The split spoon is typically driven in advance of a hollow stem auger string which allows collection of the disturbed but representative sample.

SPT sampling is performed by using a split barrel sampler in accordance with ASTM D1586. The split barrel sampler, or split spoon, consists of an 18- or 24-inch long, 2-inch outside diameter tube, which comes apart length-wise into two halves. The split spoon is typically driven in advance of a hollow stem auger string which allows collection of the disturbed but representative soil sample.

Once the borehole is advanced to the target depth and the borehole cleaned of cuttings, representative soil samples are collected in the following manner:

- the split-spoon sampler should be inspected to ensure it is properly cleaned and decontaminated. The driving shoe (tip) should be relatively sharp and free of severe dents and distortions;
- the cleaned split-spoon sampler is attached to the drill rods and lowered into the borehole. Do not allow the sampler to drop onto the soil;
- after the sampler has been lowered to the bottom of the hole, it is given a single blow to seat it and make sure that it is in undisturbed soil. If there still appear to be excessive cuttings in the bottom of the borehole, remove the sampler from the borehole and remove the cuttings; and
- mark the drill rods in three or four successive 6-inch (0.15 m) increments, depending on sampler length, so that the advance of the sampler under the impact of the hammer can be easily observed for each 6-inch (0.15 m) increment.

The sampler is then driven continuously for either 18 or 24 inches (0.45 or 0.60 m) by use of a 140-pound (63.5 kg) hammer. The hammer may be lifted and dropped by either the cathead and rope method, or by using a trip, automatic, or semi-automatic drop system. The hammer should free-fall a distance of 30 inches (±1 inches) (760 mm, ±25 mm) per blow. Measure the drop at least daily to ensure that the drop is correct. To ensure a free-falling hammer, no more than 2 1/4 turns of the rope may be wound around the cathead (see ASTM D1586). The number of blows applied in each 6-inch (0.15 m) increment is counted until one of the following occurs:

- a total of 50 blows have been applied during any one of the 6-inch (0.15 m) increments described above;

- a total of 100 blows have been applied;
- there is no advancement of the sampler during the application of ten successive blows of the hammer (i.e., the spoon is "bouncing" on a stone or bedrock); or
- the sampler has advanced the complete 18 or 24 inches (0.45 or 0.60 m) without the limiting blow counts occurring as described above.

In some cases where the limiting number of blow counts has been exceeded, the Consultant may direct the driller to attempt to drive the sampler more if collection of a greater sample length is essential.

On the field form, record the number of blows required to drive each 6-inch (0.15 m) increment of penetration. The first 6 inches is considered to be a seating drive. The sum of the number of blows required for the second and third 6 inches (0.15 m) of penetration is termed the "standard penetration resistance" or the "N-value".

Note: If the borehole has sloughed and there is caved material in the bottom, the split spoon may push through this under its own weight, but now the spoon is partially "pre-filled". When the spoon is driven the 18 or 24 inches representing its supposedly empty length, the spoon fills completely before the end of the drive interval. Two problems arise:
1. the top part of the sample is not representative of the in-place soil at that depth;
2. the SPT value will be artificially higher toward the bottom of the drive interval since the spoon was packed full. These conditions should be noted on the field log.

The sampler is then removed from the borehole and unthreaded from the drill rods. The open shoe (cutting end) and head of the sampler are partially unthreaded by the drill crew and the sampler is transferred to the geologist/engineer work surface.

Note: A table made out of two sawhorses and a piece of plywood is appropriate, or a drum, both covered with plastic sheeting.

The open shoe and head are removed by hand, and the sampler is tapped so that the tube separates.

Note: Handle each split spoon with clean disposable gloves if environmental issues are being investigated.

Measure and record the length of sample recovered making sure to discount any sloughed material that is present on top of the sample core.
Caution must be used when conducting SPT sampling below the groundwater table, particularly in sand or silt soils. These soils tend to heave or "blow back" up the borehole due to the difference in hydraulic pressures between the inside of the HSA and the undisturbed soil. To equalize the hydraulic pressure, the inside of the HSA must be filled

with water or drilling mud. The drilling fluid level within the boring or hollow-stem augers needs to be maintained at or above the in situ groundwater level at all times during drilling, removal of drill rods, and sampling. Since heave or blow back is not always obvious to the driller, it is essential that the water level in the borehole always be maintained at or above the groundwater level. Heaving conditions and the use of water or mud should be noted on the field logs.

SPT sampling below the water table in sands and silt occasionally results in low SPT values being obtained due to the heaving effect disturbing the soil especially if the water level in the hole has not been maintained at the in situ water level. Suspect low N values should be noted on the field logs. If it is critical to have accurate N values below the water table, other methods can be employed, such as conducting a dynamic cone penetration test. This quick and easy test involves attaching a cone shaped tip to the end of the drill rods, and driving the tip into the ground similar to the SPT method, except that the borehole is not pre-augered. Cones may be driven 20 to 40 feet through a formation without augering. Blow counts are recorded for each foot (0.3 m) of advancement.

A variation of split barrel sampling involves the use of a longer barrel in conjunction with hollow stem augers. The sampling barrel is installed inside the auger with a swivel attachment to limit rotation of the barrel. After completion of a 5-foot auger penetration, the auger is left in place and the barrel retrieved from the borehole. The sampler should be handled and the sample retrieved in the same way as described above for SPT sampling. This method is quicker than SPT split spoon sampling and the sample is virtually undisturbed because the cutting shoe sits ahead of the auger. No SPT information is collected due to soil sample collection during auger run.

Thin-Walled Samplers (Shelby Tubes)

Thin-walled samplers are used to collect relatively undisturbed samples (as compared to split-spoon samples) of soft to stiff clayey soils. Shelby tubes are commonly used. The Shelby Tube has an outside diameter of 2 or 3 inches and is 3 feet long. These undisturbed samples are used for certain laboratory tests of structural properties (consolidation, hydraulic conductivity, shear strength) or other tests that might be influenced by sample disturbance. Procedures for conducting thin-walled tube sampling are provided in ASTM D1587, and are briefly described below.

- the soil deposit being sampled must be cohesive in nature, and relatively free of sand, gravel, and cobble materials, as contact with these materials will damage the sampler;
- clean out the borehole to the sampling elevation using whatever method is preferred that will ensure the material to be sampled is not disturbed. If groundwater is encountered, maintain the liquid level in the borehole at or above groundwater level during the sampling operation;
- bottom discharge bits are not permitted. Side discharge bits may be used, with caution. Jetting through an open-tube sampler to clean out the borehole to sampling

elevation is not permitted. Remove loose material from the center of a casing or hollow-stem auger as carefully as possible to avoid disturbance of the material to be sampled;

- place the sample tube so that its bottom rests on the bottom of the hole. Advance the sampler into the formation without rotation by a continuous and relatively rapid motion; usually hydraulic pressure is applied to the top of the drill rods;
- determine the length of advance by the resistance and condition of the formation, but the length shall never exceed 5 to 10 diameters of the tube in sands and 10 to 15 diameters of the tube in clays;
- in no case should the length of advance be greater than the sample-tube length minus an allowance for the sampler head and a minimum of 3 inches for cuttings.
- the tube may be rotated to shear the bottom of the sample 2 to 3 minutes after pressing in, and prior to retrieval to ensure the sample does not slide out of the tube. Lift the weight of the rods off of the tube prior to rotating.
- withdraw the sampler from the formation as carefully as possible in order to minimize disturbance of the sample;
- package and transport the sample in accordance with Paragraph ix).

On occasion it maybe required to extract the sample from the tube in the field.

- a sample extruder, which consists of a clamp arrangement to hold the tube and a hydraulic ram to push the sample through the tube, is usually mounted on the side of the rig. To prevent cross-contamination, be certain that the extruder is field cleaned between each sample;
- the sample is then extruded into a carrying tray; these are often made from a piece of 4-inch or 6-inch diameter PVC pipe cut lengthwise. Be certain that the carrying tray is field cleaned between each sample. The sample is carried to the work station to describe the sample, trim the potentially cross contaminated exterior, and place it in the appropriate container; and
- the Shelby tube may then be thoroughly field cleaned and decontaminated for reuse. Since they are thin-walled, the tubes are easily damaged, crimped, or otherwise distorted during handling or pushing. The Shelby Tube should be inspected before use and any which are significantly damaged should be rejected.

v) Borehole Completion

At the completion of the soil boring, once the soil/groundwater samples have been collected, the borehole annulus is then abandoned. Borehole abandonment options are identified in SOP 2.4 - Borehole Abandonment/Sealing. Each boring will be surveyed to establish vertical/horizontal information; field ties (i.e., swing ties) will also be collected to document the boring location. Once completed, a stratigraphic log will be prepared for reporting purposes.

EQUIPMENT

- Drilling Equipment
- Stratigraphy Log (Overburden) (Form 2.6-01)
- Tape Measure

REFERENCE

1. ASTM D420-93 Guide to Site Characterization for Engineering, Design, and Construction Purposes
2. ASTM D1452-80 Practice for Soil Investigation and Sampling by Auger Borings
3. ASTM D1586-84 Test Method for Penetration Test and Split-Barrel Sampling of Soils
4. ASTM D1587-94 Practice for Thin-Walled Tube Geotechnical Sampling of Soils
5. ASTM D2488-93 Practice for Description and Identification of Soils (Visual-Manual Procedure)
6. National Water Well Association, Handbook of Suggested Practices for the Design and Installation of Ground-Water Monitoring Wells.1989
7. EPA OSWER-9950.1,1986. RCRA Ground-Water Monitoring Technical Enforcement Guidance Document

2.4 BOREHOLE ABANDONMENT/SEALING

INTRODUCTION

The following procedure describes common techniques for the abandonment/sealing of overburden boreholes. Borehole completion may have been performed by hollow stem augering/split spoon sampling, direct push sampling device, solid stem augering or other soil sample collection techniques. The method of borehole abandonment selected for a program will be dependent on a number of factors such as: depth to groundwater, presence of contamination (and degree of contamination i.e., light or dense non-agueous phase liquids - NAPL), confining layer presence and/or physical setting (i.e., open field/vacant land, vs. facility setting). The Work Plan guiding these activities (soil boring/boring closure) will dictate which method of borehole abandonment/sealing is required. The borehole abandonment/sealing techniques reviewed in the following consist of:

- soil cutting backfill;
- bentonite chip backfill; or
- cement/bentonite grout backfill using tremie techniques.

Boreholes need to be abandoned and sealed properly to prevent surface water entry to the groundwater regime, to eliminate any physical hazard, and to prevent/protect groundwater movement from one aquifer to another.

PROCEDURES REFERENCED

- 2.2 Soil Borings

BOREHOLE ABANDONMENT/SEALING PROCEDURE

A. Soil Cutting Backfill

Typically employed when working above groundwater table and at shallow depths.

- The final depth of borehole will be measured and recorded.
- Cuttings are dropped into borehole after augering/sample equipment is removed.
- Drill rod and/or probe rodding is used to compact/compress cuttings to allow return of all cuttings back into borehole.
- Mound final surface of cuttings above ground surface to allow settlements and promote surface water runoff away from boring.
- Borehole abandonment will be documented in field records/notes.

B. Bentonite Chip Backfill

Typically employed when working above or just into the groundwater table

- Excess cuttings have been drummed for disposal or excess cuttings have been spread at ground surface.
- The depth of the borehole will be measured and recorded.
- Bentonite chips will be dropped into borehole as hollow stem augers are removed, or after the boring equipment has been removed from the borehole (solid stem auger, probing tools, split spoon samplers).
- The bentonite chip backfill will be extended to within 1 foot of ground surface, the final borehole space will be backfilled with native soil and mounded slightly to allow settlement and promote surface water runoff away from the boring. Alternatively, the borehole cuttings may be mixed with bentonite to complete the abandonment/sealing task.
- Borehole abandonment will be documented in field records/notes.

C. Cement/Bentonite Grout Backfill

Typically employed when working below the groundwater table, or in an area where a confining layer exists and the potential for groundwater/NAPL movement along a preferential pathway (i.e., former borehole) must be eliminated.

- The final depth of borehole will be measured and recorded.
- The volume of grout required will be calculated from the above measurements.
- A grout mix of one bag (94 lbs.) of Portland Cement and three pounds of bentonite with approximately 7.5 gallons of clean water will be prepared.
- Using a tremie tube placed at the base of the borehole the grout will be pumped until observed at the required elevation. The tremie tube will be raised as the grout level rises (positive displacement technique).
- The bentonite/grout backfill will be extended to within 1 foot of ground surface, the final borehole space will be backfilled with native soil and mounded slightly to allow settlement and promote surface water runoff away from boring.
- Borehole abandonment will be documented, noting depth of borehole; volume of grout used and mix ratio.
- Groundwater displaced from the borehole may or may not required containment depending on borehole setting and/or water quality.

NOTE: AT THE COMPLETION OF BOREHOLE ABANDONMENT/SEALING ACTIVITIES (REGARDLESS OF METHODOLOGY EMPLOYED) IT IS NECESSARY TO CHECK FOR SURFACE SETTLEMENT A FEW DAYS AFTER WORK COMPLETION TO DETERMINE IF THE BOREHOLE AREA REQUIRES "TOPPING OFF".

Restoration

The area around the borehole shall be restored as directed by the plant representative (e.g., asphalt, concrete, vegetation).

Cleanup

The area around the borehole shall be completely cleaned up of any investigation related materials (litter, etc.).

EQUIPMENT/MATERIALS

- Grout pump/mixing equipment
- Test Boring Report

REFERENCE

1. United States Environmental Protection Agency (1992) "Guide to Management of Investigation-Derived Wastes", Quick Reference Fact Sheet
2. ASTM D5299 "Guide for Decommissioning of Ground Water Wells, Vadose Zone Monitoring Devices, Boreholes and Other Devices for Environmental Activities

2.5 SOIL CLASSIFICATION

INTRODUCTION

The stratigraphic log is a factual description of the soil at the borehole location and is relied upon to interpret the soil characteristics, and their influence and significance in the subsurface environment. The accuracy of the stratigraphic log is to be verified by the person responsible for interpreting subsurface conditions. An accurate description of the soil stratigraphy is essential for a reasonable understanding of the subsurface conditions. Confirmation of the field description by examination of representative soil samples by the project geologist, hydrogeologist, or geotechnical engineer (whenever practicable) is recommended.

The ability to describe and classify soil correctly is a skill that is learned from a person with experience and by systematic training and comparison of laboratory results to field descriptions.

PROCEDURES REFERENCED

- 2.2 Soil Borings

DESCRIPTIONS

Several methods for classifying and describing soils or unconsolidated sediments are in relatively widespread use. The Unified Soil Classification System (USCS) is the most common. With the USCS, a soil is first classified according to whether it is predominantly coarse-grained or fine-grained.

The description of fill soil is similar to that of natural undisturbed soil except that it is identified as fill and not classified by USCS group, relative density, or consistency. Those logging soils must attempt to distinguish between soils that have been placed (i.e., fill) and not naturally present; or soils that have been naturally present but disturbed (i.e., disturbed native).

It is necessary to identify and group soil samples consistently to determine the subsurface pattern or changes and non-conformities in soil stratigraphy in the field at the time of drilling. The stratigraphy in each borehole during drilling is to be compared to the stratigraphy found at the previously completed boreholes to ensure that pattern or changes in soil stratigraphy are noted and that consistent terminology is used.

Visual examination, physical observations and manual tests (adapted from ASTM D2488, visual-manual procedures) are used to classify and group soil samples in the field and are summarized in this subsection. ASTM D2488 should be reviewed for detailed explanations of the procedures. Visual-manual procedures used for soil identification and classification include:

- visual determination of grain size, soil gradation, and percentage fines;
- dry strength, dilatancy, toughness, and plasticity (thread or ribbon test) tests for identification of inorganic fine grained soil (e.g., CL, CH, ML, or MH); and
- soil compressive strength and consistency estimates based on thumb indent and pocket penetrometer (preferred) methods.

The three main soil divisions are: coarse grained soil (e.g., sand and gravel), fine grained soil (e.g., silt and clay), and soil with high natural organic matter content (e.g., peat and marl).

Coarse Grained Soil

The USCS group symbols for coarse-grained soils are primarily based on grain or particle size, grain size distribution (gradation), and percent fines (silt and clay content).

Coarse-grained soils are then further subdivided according to the predominance of sand and gravel. Course grained soil is made up of more than 50 percent, by weight, sand size, or larger (75 μm diameter, No. 200 sieve size or larger). It is noted that there are other definitions for coarse grained or coarse textured soil and for sand size such as soil having greater than 70 percent particles equal to or greater than 50 μm diameter.

Descriptions for grain size distribution of soil include; poorly graded (i.e., soil having a uniform grain size, SP and GP) and well graded (i.e., poorly sorted; having wide range of particle sizes with substantial intermediate sizes, SW and GW).

Coarse-grained soils are further classified based on the percentage of silt and clay it contains (fines content). Coarse-grained soils containing greater than 12 percent fines are commonly described as dirty. This description arises from the soil particles that adhere when the soil is rubbed between the hands or adhere to the sides of the jar after shaking or rolling the soil in the jar. The jar shake test, which results in segregation of the sand and gravel particles, is also used as a visual aid in determining gravel and sand percentages.

Examples of the group symbol, name, and adjectives used to describe the primary, secondary, and minor components of soil are; GW - Sandy Gravel (e.g., 70 percent gravel and 30 percent sand) or Sandy Gravel trace silt (less than 10 percent silt), and SP - Sand, uniform.

Relative density is an important parameter in establishing the engineering properties and behavior of coarse-grained soil. Relative density of non-cohesive (granular) soil is

determined from standard penetration test (SPT) blow counts (N values) (after ASTM Method D1586).

The SPT gives a reliable indication of relative density in sand and fine gravel. N values in coarse-grained soil are influenced by a number of factors that can result in overestimates of relative density (e.g., in coarse gravel and dilatent silty fine sand) and can be conservative and underestimate the relative density (e.g., sand below the groundwater table and uniform coarse sand). These effects will be assessed by the project manager, if required, and need not be taken into account by field personnel.

Other dynamic methods, such as modified SPT and cone penetration tests, are used on occasion to supplement or replace the SPT method for certain site-specific conditions. The details of all modifications to the SPT or substitute methods should be recorded as they are required to interpret test results and correlate to relative density.

Fine Grained Soil

A soil is fine grained if it is made up of half or more of clay and silt (i.e., fines greater than 50 percent by weight passing the 75 μm (No. 200) sieve size). A description of visual-manual field methods and criteria (after ASTM D2488) that are used to further characterize and group fine grained soil (e.g., CL, CH, ML, or MH) including dry strength, dilatancy, toughness, and plasticity (thread or ribbon test) follows. Fine-grained soils are subdivided on a basis of the liquid limit and the degree of plasticity.

The accurate identification of silts and clays can be aided by the use of some single field tests. Clay is sticky, will smear readily, and can be rolled into a thin thread even when the moisture content is low. When it is dry, clay forms hard lumps. Silt on the other hand, has a low dry strength, can be rolled into threads only at high moisture content, and a wet silt sample will puddle when it is tapped.

CRITERIA FOR DESCRIBING DRY STRENGTH

Description	***Criteria***
None	The dry specimen crumbles into powder with mere pressure of handling.
Low	The dry specimen crumbles into powder with some finger pressure.
Medium	The dry specimen breaks into pieces or crumbles with considerable finger pressure.
High	The dry specimen crumbles into powder with finger pressure. Specimen will break into pieces between thumb and a hard surface.
Very High	The dry specimen cannot be broken between the thumb and a hard surface.

CRITERIA FOR DESCRIBING DILATANCY

Description	*Criteria*
None	No visible change in small wetted specimen when rapidly shaken in palm of hand.
Slow	Water appears slowly on the surface of the specimen during shaking and does not disappear or disappears slowly upon squeezing.
Rapid	Water appears quickly on the surface of the specimen during shaking and disappears quickly upon squeezing or stretching.

CRITERIA FOR DESCRIBING TOUGHNESS

Description	*Criteria*
Low	Only slight pressure is required to roll the thread near the plastic limit. The thread and the lump are weak and soft.
Medium	Medium pressure is required to roll the thread to near the plastic limit. The thread and the lump have medium stiffness.
High	Considerable pressure is required to roll the thread to near the plastic limit. The thread and the lump have very high stiffness.

CRITERIA FOR DESCRIBING PLASTICITY

Description	*Criteria*
Nonplastic	A 1/8-inch (3 mm) thread cannot be rolled at any water content.
Low	The thread can barely be rolled and the lump cannot be formed when drier than the plastic limit.
Medium	The thread is easy to roll and not much time is required to reach the plastic limit. The thread cannot be re-rolled after reaching the plastic limit. The lump crumbles when drier than the plastic limit.
High	It takes considerable time rolling and kneading to reach the plastic limit. The thread can be re-rolled several times after reaching the plastic limit. The lump can be formed without crumbling when drier than the plastic limit.

Examples of group symbol identification based on visual-manual procedures and criteria for describing fine grained soil are:

Group Symbol	***Dry Strength Plasticity***	***Dilatancy***	***Toughness***
ML	None to low	Slow to rapid	Low or thread cannot be formed
CL	Slight Medium to high	None to slow	Medium
MH	Low Low to medium	None to slow	Low to medium Low
CH	High to very high	None	High High

A requirement for positive classification by USCS group symbols (as described in Test Method ASTM D2487) is laboratory determination of particle size characteristics, liquid limit and plasticity index. The need for this type of testing will be determined by the project geologist, hydrogeologist, or geotechnical engineer.

Examples of name terminology that accompanies the group symbols are ML - Sandy Silt (e.g., 30 percent sand) and CL - Lean Clay with sand (e.g., 15 to 29 percent sand).

The correlation between N value and consistency for clays is rather unreliable. It is preferable to determine consistency using more appropriate static test methods, particularly for very soft to stiff clay soil. N value estimates of consistency are more reasonable for hard clay.

Unconfined compressive strength (Su) may be estimated in the field from the pocket penetrometer test method. To obtain a pocket penetrometer estimate of consistency and compressive strength, the soil core is cut perpendicular to the core length, the length of core (minimum 4 inches) is held in the hand and a moderate confining pressure is applied to the core (not sufficient to deform the core); the penetrometer piston tip is slowly inserted into the perpendicular face of the core until the penetrometer indents into the soil core to the mark indicated on the tip of the penetrometer piston; the penetrometer estimate of soil compressive strength (Su) is the direct reading of the value mark on the graduated shaft (in tons per square foot or other unit of pressure as indicated) indicated by the shaft ring marker, or in some models, by the graduated piston reading at the shaft body. To obtain an average estimate, this procedure is completed several times on both ends and mid cross-section of the core. For Shelby Tube (or thin wall sampler) samples the pocket penetrometer tip is applied to the exposed bottom of the sample at several locations.

Estimates of compressive strength for clay soil of very soft to stiff consistency are better established by in situ shear vane tests or other static test methods.

The description of consistency (or strength) is an important element in determining the engineering properties and strength characteristics of fine-grained cohesive soil. Consistency terms (e.g., soft, hard) are based on the unconfined compressive strength (Su) and shear strength or cohesion (cu) of the soil.

The ease and pattern of soil vapor and groundwater movement in the subsurface is influenced by the natural structure of the soil. Soil structure, for the most part, depends on the deposition method and, to a lesser extent, climate.

Visual Appearance/Other Features

Those logging soils should also note the presence, depth and components of fill soils (if evident), and note the distinction between disturbed native soils (i.e., excavation likely performed) vs. undisturbed native soils.

Other features such as root presence/structure, and soil fractures should also be recorded. Soil fractures should be described noting fracture orientation (i.e., horizontal/vertical), length/aperture and appearance of soil infilling, oxidation and/or weathering (if present).

FIELD SCREENING

Field Sample Screening

Upon the collection of soil samples, the soil is screened with a photoionization detector (PID) for the presence of organic vapor. This is accomplished by running the PID across the soil sample. The highest reading and sustained readings are recorded.

Note: The PID measurement must be done upwind of the excavating equipment or any running engines so that exhaust fumes will not affect the measurements.

Another method of field screening is head space measurements. This consists of placing a portion of the soil sample in a sealable glass jar, placing aluminum foil over the jar top, and tightening the lid. Alternatively, plastic sealable bags maybe utilized for field screen in lieu of glass containers. The jar should only be partially filled. Shake the jar and set aside for at least 30 minutes. After the sample has equilibrated, the lid of the jar can be opened; the foil is punctured with the PID probe and the air (headspace) above the soil sample is monitored. This headspace reading on the field form or in the field book is recorded.

Note: Perform all headspace readings in an area that is not subject to wind. Also, in the winter, it is necessary to allow the samples to equilibrate in a warm area (e.g., site trailer, van, etc.). This requirement is dictated by the Work Plan.

All head space measurements must be completed under similar conditions to allow comparability of results.

NAPL DETECTION

During soil examination and logging, the sampler shall carefully check for the presence of light or dense NAPL. NAPL may be present in gross amounts or present in small/minute quantities. The adjectives and corresponding quantities used when describing NAPL within a soil matrix are as follows:

Visual Description	***Fraction of Soil Pore Volume Containing NAPL***
Saturated	>0.5
Some	0.5 - 0.25
Trace	<0.25

A complete description of NAPL, must describe the following:

- color;
- quantity;
- density (compared to water i.e., light/floats or heavy/sinks);
- odor (if observed); and
- viscosity (i.e., mobile/flowable, non-mobile/highly viscous-tar like).

The presence of an "iridescent sheen" by itself does not constitute 'NAPL presence', but may be an indicator that NAPL is close to the area.

NAPL presence within a soil matrix may be confirmed by placing a small soil sample within water, shaking, and observing for NAPL separation (i.e., light or dense), from the soil matrix.

Trace amounts of NAPL are identified/confirmed by a close visual examination of the soil matrix, [i.e., separate soil by hand (wearing disposable gloves)] and careful inspection of the soil separation planes/soil grains is performed for NAPL presence.

Often during the sample examination with a knife, an iridescent sheen will be noted on the soil surface (i.e., clay/silts) if the knife has passed through an area of NAPL.

There are a number of more sophisticated tests available to confirm/identify NAPL presence, these are:

- UV fluorescent analysis;
- Sudan IV dye shake test;
- hydrophobic dyes;
- centrifugation; and
- chemical analysis.

For UV fluorescence, soil samples are placed in a Ziploc bag, which are marked with the boring location and depth. The samples are then taken to a dark room where they are placed under a UV light. Samples with DNAPL present in them will fluoresce a milky-white color.

Sudan IV dye shake tests are performed by combining a small amount of soil sample, water and Sudan IV together in a vial with a cap. The Sudan IV is fat-soluble, thus, the dye will be released to the water in the presence of DNAPL. After combining the soil, water and dye, cap the vial and shake vigorously. If DNAPL is present, the water will become a pinkish-red hue.

The Sudan IV dye shake test is dependent upon the nature of the soil that is being tested. Clayey soils have a tendency to cloud the water and obscure the dye release. If clayey soils are the target zone, a combination of visual observation, field and headspace screening, UV fluorescence and Sudan IV dye shake tests should be performed to determine whether DNAPL is present in the soil.

Typically consultants will utilize organic vapor detection results, visual examination, soil/water shake testing, and chemical analysis, to confirm NAPL presence. The more complex techniques described may be incorporated on sites where clear colorless NAPL is present and its field identification is critical to the program.

Note: When describing the presence of vegetative matter in the soil sample, do not use the term "organic" as this often leads to confusion with regards to the presence of organic chemicals (i.e., NAPL).

EQUIPMENT/MATERIALS

- Pocket knife or small spatula
- Small handheld lens
- Stratigraphic Log (Overburden) (Form 2.6-01)
- Tape Measure

- Sudan IV dye
- Vial
- Ziplock bags

REFERENCES

1. American Society for Testing and Materials (1991), Standard D1452-80, "Practice for Soil Investigation and Sampling by Auger Borings", Annual Book of ASTM Standard, Section 4, Volume 04.08.
2. ASTM Standards on Environmental Sampling (1995), Standard D 2488-93, "Standard Practice for Description and Identification of Soils (Visual-Manual Procedure)"
3. ASTM Standards on Environmental Sampling (1995), Standard D 4700-91, " Guide for Soil Sampling from the Vadose Zone".
4. ASTM Standards on Environmental Sampling (1995), Standard D 1586-92, " Test Method for Penetration Test and Split-Barrel Sampling of Soils".
5. ASTM Standard D 2487, " Classification of Soils for Engineering Purposes (Unified Soil Classification System)".
6. Geotechnical Gauge, Manufactured by W.F. McCollough, Beltsville, MD.
7. Sand Grading Chart, by Geological Specialty Company, Northport, Alabama.

2.5 PROCEDURES FOR SOIL VAPOR MONITORING POINT INSTALLATION

INTRODUCTION

This document includes procedures for the installation of a soil vapor monitoring point intended for the collection of subsurface soil gas samples.

DRILLING PROCEDURES

Operating Procedures 2.1 – Drilling Techniques presents descriptions of various drilling methods that are available, however, due to the relatively shallow monitoring point depth and small diameter (i.e. screen diameter and associated tubing) typically only direct-push methods and hand auger methods (depending on Site access and soil type) apply.

- Utility clearing will be completed in accordance with *Operating Procedure 1.1 - Utility Clearance*.

- Drilling/boring equipment will be decontaminated prior to the installation of the soil vapor point and between successive installations in accordance with *Operating Procedure 7.0 – Equipment Decontamination.*

- No oils or grease will be used on equipment introduced into the borehole.

- Derived waste generated from boring activities will be characterized and disposed of in accordance with *Operating Procedure 8.0 – Waste Characterization.*

- The target depth interval shall be pre-determined by the site-specific Project Work Plan but may be amended based on field observations of the underlying stratigraphy/geology. The bottom of the probe hole should be kept at least 1 foot above the expected water table to ensure that groundwater or water from the capillary fringe does not intersect the sand pack of the probe. Sample collection intervals should be determined by site-specific project sampling objectives. Depending on the type of tooling utilized by a direct-push drilling rig (e.g., Geoprobe) or hand-operated soil vapor sampling kit (e.g. AMS GVP Kit), borehole diameters could range from 1.25 to 2.25 inches.

- Soil classification will be completed in accordance with *Operating Procedure 2.6 – Soil Classification*.

- Special attention should be paid to the moisture content of soils throughout the soil profile. Observations of soil moisture should be noted on the well log for each

boring. If soil moisture data are not available, it may be helpful to collect soil samples during drilling for laboratory determination of soil moisture content adjacent to the probe screen.

SOIL VAPOR POINT INSTALLATION

Once the target depth has been reached, ¼-inch O.D. tubing (stainless steel, Teflon, or nylon) will be connected to a stainless steel screen implant with a minimum length of 6-inches that is designed for soil gas sampling, and deployed down the borehole. The screen implant is connected to one end of the sample tubing and the associated tubing is placed on the inside of the probe rod up to the surface during placement (Note: the tubing should be temporarily capped with a fitting made of inert materials, such as brass or nylon). Depending on soil type (e.g., dry sands with very little fines), the borehole may easily collapse requiring the use of a temporary outer casing (typically a larger diameter probe rod) large enough to allow deployment of the stainless steel screen implant and attached tubing down the temporary casing once the bottom of the casing is in place and at the targeted depth (see Figure 1).

The temporary casing probe rod is slowly removed during the placement of filter pack sand and the bentonite annular seal. Clean, uniform, well-rounded, silica sand should be placed as the filter pack in the annular space around the screen and casing from the base of the screen to approximately 6-inches above the top of the screen implant. The stainless steel screen implants require filter pack silica sand with a particle size larger than the pore size of the screen implant (greater than 0.0057 inch or 0.145 mm).

Granular or flaked bentonite will then be added from the top of the filter pack to approximately 6-inches below ground surface (bgs) to allow room for the flush mount cover. To insure an air tight annular seal, a small amount of distilled water should be added throughout the bentonite placement. (Caution: over hydration of the bentonite could lead to water in the screened interval which may compromise the use of the soil vapor point.) A weighted tape or other measuring device can be utilized to provide accurate measurements and to avoid bridging during the placement of filter pack sand and bentonite.

After the construction of the annular seal, an appropriate sized flush mount cover will be installed to protect the soil vapor point. Pre-mixed concrete will be used to secure the flush mount cover in place and to provide a sufficient surface seal for the soil vapor point. Although a 4-inch flush mount cover is typically sufficient for either indoor or outdoor soil vapor point installations, site conditions may warrant use of other materials (e.g., larger diameter cover, traffic rated cover). Details regarding the type of appropriate well covers and concrete surface seals are similar to those found in most monitoring well installations and are discussed in *Operating Procedure 3.1 – Well Construction Materials.*

At the completion of the vapor point installation, the temporary tubing cap will be removed and the tubing will be equipped with a gas tight brass or nylon valve near the end of the tubing. If sampling with a Summa® canister is planned, the end of the tubing can be fitted with a brass or stainless steel Swagelok® (or similar) compression fitting, as long as the fitting is compatible with the canister and flow controller connections.

Upon completion of the installation of the sampling point, valve, and fittings, the volume of air in the tubing and sand pack will be calculated. Approximately three (3) times the calculated volume of air will be slowly (<100 ml/Min) purged immediately after installation and sealing of the sampling point. Samples should not be collected for 24 to 48 hours after installation of the sampling point and purging of the sand pack. Purging will be accomplished using a large graduated syringe, pre-calibrated personnel air sampling pump, hand-operated vacuum pump, or a Summa® canister that has been designated for purging only. Purge volumes should be recorded and included in the reporting for the installation of the soil gas monitoring point.

EQUIPMENT/MATERIALS

The following equipment and materials are used for the completion of a soil vapor monitoring point:

- Appropriate personal protective equipment (PPE) as required by the site specific Health and Safety Plan.
- Site Plan, Field Sampling Plan, and/or Work Plan, with proposed soil vapor monitoring point locations.
- Drilling/boring equipment appropriate for the Site and in certain instances dictated by the proposed location.
- Soil vapor monitoring point materials including; tubing, vapor screen implant, silica sand filter pack, bentonite seal material, distilled water, flush mount well cover, concrete mix, and associated fittings and connections for capping the tubing and securing the vapor point. (Materials are further defined throughout this Operating Procedures.)
- Weighted tape measure.
- Purge pump calibrated to remove initial ambient air within tubing and borehole.

WASTE MANAGEMENT

The waste materials generated by these activities should be minimal. Personal protective equipment, such as gloves, disposable clothing, and other disposable equipment will be disposed of in accordance with Operating Procedure 8.0 – Waste Characterization.

REFERENCE

1. American Society for Testing and Materials (1991), Standard D1452-80, "Practice for Soil Investigation and Sampling by Auger Borings", Annual Book of ASTM Standard, Section 4, Volume 04.08.

2. American Society for Testing and Materials (1991), Standard D2113-83 (87), "Diamond Core Drilling for Site Investigations", Annual Book of ASTM Standards, Section 4, Volume 04.08.

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4. API, 2004. Collecting and Interpreting Soil Gas Samples from the Vadose Zone: A Practical Strategy for Assessing the Subsurface-Vapor-to-Indoor-Air Migration Pathway at Petroleum Hydrocarbon Sites. American Petroleum Institute, Final Draft, October, 2004.

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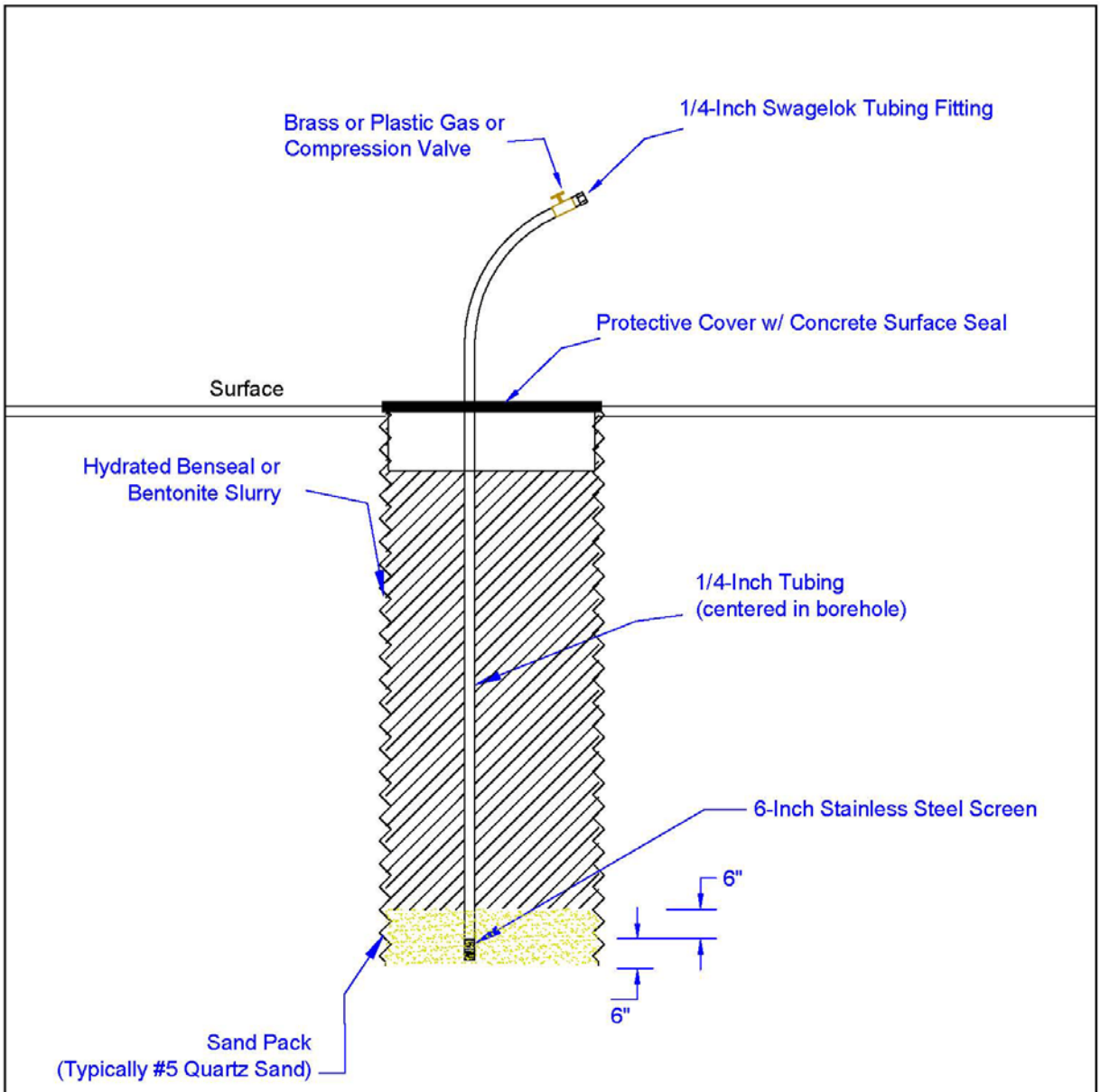
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7. Environmental Protection Agency (1988), Guidance for Conducting Remedial Investigations and Feasibility Studies Under CERCLA, Interim Final, EPA/540/G-89/004.

8. Hartman, 2004. How to Collect Reliable Soil-Gas Data for Risk-Based Applications – Specifically Vapor Intrusion. Part 3 – Answers to Frequently Asked Questions. LUSTLine Bulletin 48, November 2004.

9. State of California, 2003. State of California Advisory – Active Soil Gas Investigations. Department of Toxic Substances Control and California Regional Water Quality Control Board, Los Angeles Region. January 28, 2003

10. State of California. 2005. Interim Final: Guidance for the Evaluation and Mitigation of Subsurface Vapor Intrusion to Indoor Air. Department of Toxic Substances Control, California Environmental Protection Agency, December 15, 2004 (Revised February 7, 2005).



TYPICAL SOIL VAPOR MONITORING POINT
CONSTRUCTION DETAILS

FIGURE
1

3.0 MONITORING WELLS

3.1 WELL CONSTRUCTION MATERIALS

INTRODUCTION

In environmental subsurface investigations, the information used to evaluate subsurface conditions often relies on the installation of appropriate groundwater monitoring wells. The application and use of the proper well construction materials to the specific well installation is crucial to obtaining representative and reliable groundwater samples.

The two general types of wells are groundwater monitoring wells and pumping (also referred to as recovery, extraction, or withdrawal) wells. The specific use of a groundwater well dictates the types of materials with which it is constructed.

This SOP outlines the general types and use of well construction materials and considerations involved in selecting appropriate materials for specific well installation applications. Installation of these materials is described in detail in the specific well-installation SOPs listed below.

PROCEDURES REFERENCED

- 3.2 Overburden Monitoring Well Installation
- 3.3 Top of Bedrock Well Installation
- 3.4 Deep Bedrock Well Installation

MATERIAL DESCRIPTIONS

A. Well Screen

The screen is the portion of the well that contains appropriately sized openings and allows groundwater to enter the well. The screen materials used in groundwater monitoring wells are crucial to ensuring the installation of an efficient, productive, and durable groundwater well.

The diameter of the well screen is generally dependent upon the application of the well. For monitoring wells used in groundwater level measurements and groundwater sampling, screen diameter will generally be 2.0-inch inner-diameter (I.D.) flush-threaded screen segments (piezometers are typically 1.0-inch inner diameter but may also be 2-inch). These screen segments are typically available in 10-foot lengths. Four-inch diameter or larger well screens are usually used for recovery or production well

applications where larger diameters permit greater groundwater withdrawal rates. Larger diameter wells also allow a well to serve additional functions such as housing extraction oil recovery systems.

Screen material will be either thermoplastic Schedule 40 PVC (PolyVinylChloride) (ASTM D1785, ASTM D2665, ASTM F480) or Schedule 5 Type 316 stainless steel, depending primarily on the depth of the well and the groundwater quality (degree and nature of contamination). Shallower depths and generally low levels of contaminants in groundwater allow for PVC applications, whereas greater depths and severely degraded groundwater quality, or the presence of free-phase oils or solvents, may necessitate stainless steel due to its greater strength and resistance to chemical degradation. It should be noted that PVC and stainless steel are appropriate for the vast majority of environmental applications, and are generally accepted by regulatory agencies. Well materials other than PVC or stainless steel should be used only in certain instances, to be determined and approved by the Consultant Project Manager on a case-by-case basis.

Certain applications such as investigation of inorganic (metals) concentrations in groundwater, or the presence of low pH (acidic) conditions may preclude the use of stainless steel wells. Stainless steel, which contains molybdenum in addition to its iron content, may leach out metal compounds which could cause to misleading groundwater analysis results.

PVC may likewise leach out or degrade specific thermoplastic elements of its composition which may compromise the well integrity or groundwater analyses. PVC generally performs well in acidic groundwater conditions; however, it may degrade in the presence of certain organic compounds such as ketones, aldehydes or chlorinated compounds in high concentrations. Certain additives to the PVC may also affect groundwater quality.

Well screen slot sizes and well screen type will also be consistent for groundwater monitoring wells. Screen slot size is typically 0.010 inches; 0.020-inch slot size may be more appropriate for coarser formation materials or where the well may serve as a recovery well for free-phase oils. For monitoring applications, slot type should be either factory machine-slotted or continuous-wrap slotted. Perforated, bridge-slotted or louver-slotted well screens are generally not acceptable for most environmental applications and should be avoided.

Screen slot sizes may vary from these two sizes when used in production or recovery (pumping) well applications where the need to maximize groundwater withdrawal is essential. In such cases, screen slot sizes can be manufactured to exact specifications for a particular well based on particle size analysis results and formation transmissivity or permeability.

B. Well Riser Pipes and Casings

Well riser pipe is a solid extension of the well that extends from the screen up to the surface. The riser pipe protects the well screen, prevents outside groundwater from entering the well, and allows groundwater pumped from down in the open interval to be routed up through the well to the surface.

Well riser pipe should be of the same material and size as the well screen described above. In instances to be determined and approved by the project manager on a case-by-case basis only, differing materials may be approved for use in the same well (e.g. stainless steel well screen connected to PVC riser). Well risers should extend to the surface and should either be cut at grade in flush-mount completions or as an approximately 3-foot stickup to be covered with a steel protective casing.

Well riser pipe sections shall be flush-threaded and fitted with neoprene, rubber, or other appropriately constructed, durable o-rings to properly seal the threaded pipe joints. Glues or cements are not to be used in well construction.

In installations of bedrock monitoring wells, which have an open rock monitoring interval and a permanent well casing that extends from bedrock to the surface, the permanent casing (or casings in telescoping wells) shall be made of carbon steel, low-carbon steel ($\geq$ 0.8% carbon and $\leq$ 0.8% carbon, respectively), or PVC. The well casing should be a minimum of 4 inches in diameter (at least 4-inch diameter for the innermost casing).

C. Sand Packs

The filter pack, or sand pack, installed in a well replaces formation material immediately around a well with a more permeable material (sand). The sand pack separates the well screen from the formation, increases the hydraulic diameter of the well, and prevents fines (silt or clay) from entering or clogging the well screen.

Sand pack of an appropriate size shall be utilized based on the well screen slot size being used. Sand pack size should be chosen so that the majority of the sand (sand pack has inherent variation in its particle grain size distribution) is larger than the screen slot size while sized small enough to prevent deleterious amounts of formation fines from entering the well through the sand pack. Screen slot sizes of 0.010-inch and 0.020-inch typically use a sand pack such as Morie or U.S. Silica #1, #0, #00N, or equivalent.

Sand pack shall be washed silica sand with a silica content of at least 95%. Sands should meet one or more of the following requirements: NSF Standard 61, AWWA B-100, ANSI, or equivalent standards for uniformity and chemical inertness. In cases to be determined and approved by the project manager on a case-by-case basis only, differing sand pack materials may be approved for use in a well. Sand packs used for production and recovery wells with larger screen slot sizes will use larger-particle-sized sand packs

of the same type and quality. The slot size and sand pack size for recovery wells should be chosen based on results of formation grain size distribution analysis.

D. Seals

Bentonite and grout seals are installed above the sand pack to isolate the monitoring interval and prevent groundwater from infiltrating into the well screen from other water-bearing zones. Seals also prevent migration of backfill or formation materials downward into the sand pack.

Bentonite is the generic name for a group of a naturally-occurring clay minerals (montmorillonites) that come in a variety of forms: pellets, chips, granulated, or powdered. This material is commercially available as "Wyoming Bentonite". When hydrated it swells to many times its original volume and forms an ultra-low-permeability clay seal.

Bentonite chips or pellets are generally used to create a seal immediately above the sand pack. The chips/pellets are dropped inside the augers or well casing by hand down through the water column onto the top of the sand pack. Care must be taken to prevent "bridging" of the bentonite particles in the casing above the target zone. Measurements of the depth to the top of the seal must be obtained during installation of the seal to ensure its proper position and thickness. In the absence of significant water in a casing or borehole, potable water must be added to hydrate the bentonite. The bentonite seal will be allowed to set for a minimum of one-half hour, in order to hydrate properly, before additional seals (grout) are applied. Once the bentonite has set for one-half hour the grout seal may be placed, as described below.

In saline groundwater environments, such as where ocean water may infiltrate the monitoring interval, a zeolite-based seal material may be used, as saline conditions may hamper the performance of bentonite pellets.

Portland cement grout (grout) forms a concrete-like seal that can be more manageable than bentonite (e.g. able to be pumped through a water pump). Grout is generally placed on top of the hydrated bentonite seal to form a solid cement seal around the well riser up to the surface.

The grout mixture will consist of one 94-pound bag of Portland Cement and 3 to 5 pounds of powdered bentonite added per sack of cement. Two (2) pounds of calcium chloride may also be added (under certain conditions, e.g. very cold days) to accelerate the setting time of the grout, as well as to increase the dry strength of the grout. The grout will be thoroughly mixed with 6.5 gallons of potable water per sack of cement. Grout is generally placed using either the tremie or Halliburton grouting methods. These are described in the specific well installation SOPs.

E. Protective Casings and Surface Seals

Once the well screen, riser, and all seals have been placed to ground surface, the well riser must be protected. This includes protection from vehicles, damage, surface water infiltration, and weather. This is typically accomplished using either a flush-mount roadbox or a stickup casing.

Flush-mount roadboxes are circular steel casing segments with a heavy-duty steel lid with locking bolts. These units are widely available and come in a number of diameters and lengths, depending on the well diameter. A stickup protective casing is generally a length of carbon or stainless steel pipe with a locking top.

For a typical 2-inch monitoring well, the roadbox should be at least 6 inches in diameter; a stickup casing should be at least 4 inches in diameter. A roadbox should be at least 12 inches in length (they are typically 16 to18 inches long) and is installed flush with the ground surface. A stickup casing should be at least 5 to 6 feet long such that approximately 2.5-3 feet is below ground surface and 2.5-3 feet is protruding above grade. In wells where a permanent steel casing is installed (serves as the well riser pipe) and brought to the ground surface, it may be used as the protective casing provided it is equipped with a semi-permanent, metal, locking cap or cover that can be affixed to the steel casing.

Flush-mount installations should have at least the last 18-inches of the open borehole filled with coarse sand, placed up to ground surface to allow drainage of surface water infiltration down through and out of the roadbox. This also prevents infiltrating surface water from accumulating up over the top of the well riser and draining down into the well. This sand drain is not necessary in the locking-cap stickup casings.

Both roadbox and stick-up casings must be secured in the ground with concrete, which also serves as a surface seal.

In areas of high vehicle traffic activity, protective steel bollards should be installed. This is typically a vertically-oriented, concrete-filled, steel pipe (min. 4-in diameter) cemented at least 3 ft. into the ground, acting as a “guard rail” for the well casing and preventing it from being damaged by vehicles. Three bollards should be placed around a well to provide adequate protection.

EQUIPMENT

- Drilling equipment
- Well screen and riser materials
- Sand pack

- Bentonite pellets/chips
- Powdered bentonite
- Portland cement

REFERENCE

1. ASTM D1785-99, Standard Specification for Poly(Vinyl Chloride) (PVC) Plastic Pipe, Schedules 40, 80, and 120.

2. ASTM D2665-00, Standard Specification for Poly(Vinyl Chloride) (PVC) Plastic Drain, Waste, and Vent Pipe and Fittings.

3. ASTM F480-00, Standard Specification for Thermoplastic Well Casing Pipe and Couplings Made in Standard Dimension Ratios (SDR), SCH 40 and SCH 80.

4. ASTM A53/A53M-01 Standard Specification for Pipe, Steel, Black and Hot-Dipped, Zinc-Coated, Welded and Seamless for Ordinary Uses.

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3.2 PROCEDURES FOR OVERBURDEN MONITORING WELL INSTALLATION

INTRODUCTION

This procedure presents the drilling, installation, and completion of overburden monitoring wells. The design and installation of monitoring wells involves the drilling of boreholes into various types of geologic formations. Designing and installing monitoring wells may require different drilling methods and installation in the overburden. However, due to specific site and monitoring requirements, the procedure may be revised as necessary to reflect these site-specific needs. Prior planning is required for the selection of the appropriate drilling method and selection of well materials.

PROCEDURES REFERENCED

- 2.5 Soil Classification
- 3.6 Well Development
- 5.1 Soil Sampling
- 7.0 Equipment Decontamination
- 8.0 Waste Characterization

DRILLING PROCEDURES

- Drilling and sampling equipment arriving on site will be decontaminated prior to drilling in accordance with the Decontamination and Waste Disposal Procedure.
- Drilling generated waste materials will be disposed according to the Decontamination and Waste Disposal Procedure.
- Record construction and geologic information on the attached subsurface boring log.

- The depth to the target interval may be determined from an existing adjacent monitoring well/boring or from information obtained from split spoon sampling of the borehole. The criteria for determining the target interval to be monitored will be presented in the Project Work Plan. An 8-inch diameter borehole will be advanced to the target interval using a minimum 4¼-inch inside diameter (I.D.) hollow-stem auger.

- In the instances where the borehole is advanced deeper than the target interval, a bentonite pellet seal will be installed to bring the bottom of the boring to within 6 inches of the target interval. Six inches of filter sand will then be placed above the bentonite seal prior to further well installation.

- In some areas where the water table is known to be at or near the top of bedrock, the base of the overburden well will be installed at the top of bedrock.

- Continuous split-spoon samples may be collected, if required by applicable project Work Plan. (see Subsurface Soil Sampling Procedure). If collected, soil samples will be classified in accordance with the Soil Classification Procedure.

WELL CONSTRUCTION

- The well construction procedures presented below are the recommended standards. However, due to variations in subsurface conditions, changes in these well construction standards may be necessary in order to facilitate the installation of the protective casing.

- Overburden wells will be constructed of either 2-inch Schedule 40 Flush-threaded Virgin PVC or type 316 Stainless Steel. Type of well material to use will be dependent upon known subsurface conditions. Wells constructed of PVC are preferable; however in situations where sufficient levels of chlorinated solvents are present to affect the PVC well integrity, stainless steel will be the material of choice. Wells of mixed construction materials are not acceptable. The well screen will consist of machine slot or continuous wrap PVC or Stainless Steel with screen slot size appropriate for the type of subsurface material. It is anticipated that PVC will be used at the site.

- The bottom of the well screen will be placed to the bottom of the borehole. Ideally, the top of the well screen should be greater than 4 feet below grade. As the augers are slowly removed, clean washed silica sand filter pack will be placed in the annular space around the well screen and casing from the base of the screen to at least 2 feet above the screen.

- In wells that exhibit a water table elevation above the sand pack, a minimum of 2-foot thick layer of bentonite pellets will be placed above the sand pack. The seal will be hydrated and allowed to set for approximately 30 minutes.

- Cement/bentonite grout will be placed from the top of the bentonite seal to a point 5 feet below existing ground surface where conditions allow. The grout will be prepared in the ratio of one bag (94 pounds) of Type I or Type II Portland Cement to 3 to 5 pounds of bentonite powder mixed with approximately 7 gallons of potable water.

- Accurate measurements of the material depths will be made by sounding the annulus during installation. The volume of materials needed will be calculated and compared to the actual volume used. Material depths will be recorded on the well installation report log (attached).

- The well casing will be secured with a vented lockable cap. If the well is located in a high traffic area, the casing will be protected by 9-inch flush-mounted roadway box set in a concrete seal. Alternatively, in low traffic areas, the well casing may be cut above grade and completed with 4 or 6-inch diameter steel protective casing with approximately 3 feet of stick up, set in a concrete surface seal.

- For flush-mounted wells, a 9-inch diameter, water-tight protector will be installed complete with a sand drain. A lockable gripper plug will top the PVC casing.

- After installation, the monitoring well will be labeled with the well identification and a reference point for water level and depth measurements will be notched into the well casing. The well will be allowed to sit for at least 24 hours prior to well development, and for one week between development and groundwater sampling.

EQUIPMENT

- Drilling Equipment
- Well Supplies
- Subsurface Boring Log
- Overburden Well Log
- Tape Measure

REFERENCE

1. American Society for Testing and Materials (1991), Standard D1452-80, "Practice for Soil Investigation and Sampling by Auger Borings", Annual Book of ASTM Standard, Section 4, Volume 04.08.

2. American Society for Testing and Materials (1991), Standard D2113-83 (87), "Diamond Core Drilling for Site Investigations", Annual Book of ASTM Standards, Section 4, Volume 04.08.

3. American Society for Testing and Materials (1991), Standard D5092, "Practices for Design and Installation of Ground Water Monitoring Wells in Aquifers", Annual Book of ASTM Standard, Section 4, Volume 04.08.

4. New York State Department of Environmental Conservation (1988), Draft Generic Environmental Impact Statement on the Oil, Gas, and Solution Mining Regulatory Program, Division of Mineral Resources.

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3.3 WELL DECOMMISSIONING PROCEDURES

INTRODUCTION

This procedure is for the decommissioning/abandonment of groundwater monitoring wells that have been installed in overburden, top of bedrock, or deep bedrock formations. Well decommissioning refers to the procedure used to properly abandon or remove the monitoring well from the formation while taking the proper precautions to help eliminate cross-contamination.

The methods for properly abandoning monitoring wells are either by leaving the well materials in place and pressure grouting with a cement/bentonite slurry directly into the well or by over-drilling with augers, removing the well material and back filling with a cement bentonite slurry.

PROCEDURES REFERENCED

- 3.2 Procedures for Overburden Well Installation
- 7.0 Equipment Decontamination
- 8.0 Waste Characterization

WELL DECOMMISSIONING PROCEDURES

1. Pressure Grouting

- The borehole log from the monitoring well needs to be obtained to determine the well construction in order to prepare the proper materials and calculate the quantity of cement/bentonite slurry that will be required.
- The cement pad and the well protector around the monitoring pad needs to be removed and the immediate area around the monitoring well dug out. The riser pipe is to be cut off approximately one to two-feet below ground surface.
- A tremie pipe will be placed into the well completely to the bottom. A cement/bentonite slurry will then be pressure grouted in to the monitoring well backfilling completely to the surface. The grout will be prepared in the ratio of one bag (94 pounds) of Type I or Type II Portland Cement to 3 to 5 pounds of bentonite powder mixed with approximately 7 gallons of potable water. The grout

will be allowed to sit for approximately one hour to allow any settlement of the cement/bentonite slurry and then augment if needed.

2. Overdrilling

- Based on the diameter of the monitoring well, this information can be obtained from the well completion diagram, the proper sized augers need to be specified.

- The cement pad and the well protector around the monitoring pad needs to be removed and the immediate area around the monitoring well dug out. The riser pipe is to be cut off approximately one to two-feet below ground surface.

- The augers are then placed over the riser pipe of the monitoring well and then drilling commences. The drilling continues until the final depth to which the monitoring well was installed is reached. The well materials are then removed (pulled) from the augers.

- A Cement/bentonite grout will be placed from the bottom of the borehole to the top of the augers. As each flight of augers is removed from the ground, the cement/bentonite grout will continue to be placed in the augers, to the top. This will continue until all the augers have been removed from the borehole. The grout will be prepared in the ratio of one bag (94 pounds) of Type I or Type II Portland Cement to 3 to 5 pounds of bentonite powder mixed with approximately 7 gallons of potable water.

- Documentation/Notification requirements include modification of the well log to reflect closure and if necessary notification to the appropriate regulatory agency.

WASTE DISPOSAL

- All waste generated will be disposed in accordance to the methods and procedures contained in the Waste Characterization SOP.

- All material generated during well decommissioning procedures will be collected and contained on site in roll-off boxes or 55-gallon drums for future analysis and appropriate disposal.

- Personal protective equipment, such as gloves, disposable clothing, and other disposable equipment, resulting from personnel cleaning procedures and from soil sampling and handling activities, will be placed in plastic bags. These bags will be transferred into appropriately labeled 55-gallon drums or a covered roll-off box for appropriate disposal.

EQUIPMENT

- Drilling Equipment
- Well Supplies
- Monitoring Well Abandonment Form
- Tape Measure

REFERENCE

1. Michigan Department of Public Health, Ground Water Quality Control Section – Division Of Water Supply (1988), Michigan Water Well Grouting Manual, MDPH GW-3-302

2. ASTM D5229, "Guide for Decommissioning of Groundwater Wells, Vadose Zone Monitoring Devices, Boreholes and other Devices for Environmental Activities".

3.4 WELL DEVELOPMENT PROCEDURES

INTRODUCTION

This procedure is for the development of groundwater monitoring wells that have been installed in overburden, top of bedrock, or deep bedrock formations. Before a newly constructed well can be used for water-quality sampling, measuring water levels, or aquifer testing, it must be developed. Well development refers to the procedure used to clear the well and formation around the screen of fine-grained materials (sands, silts, and clays) produced during drilling or naturally occurring in the formation.

Well development is completed to remove fine-grained materials from the well but in such a manner as to not introduce fines from the formation into the sand pack. Well development continues until the well responds to water level changes in the formation (i.e., a good hydraulic connection is established between the well and formation) and the well produces clear, sediment-free water to the extent practical.

PROCEDURES REFERENCED

- 3.2 Procedures for Monitoring Well Installation
- 8.0 Waste Characterization

WELL DEVELOPMENT PROCEDURES

- The well development procedures presented below are the recommended standards. However, due to variations in conditions, changes in these well development standards may be necessary in order to facilitate the successful completion of developing the monitoring well. Well development can be accomplished by using in-place pumps or using manual equipment; either peristaltic, bladder, or other appropriate pumps depending on well depth.

- Don appropriate safety equipment.
- Attach appropriate pump and lower tubing into well.
- Turn on pump. If well runs dry, shut off pump and allow to recover.
- Surging will be performed by raising and lowering the pump in the well to open and close the check valve in the pump several times to pull fine-grained material from the well. Collect the groundwater sample in a glass jar to determine relative turbidity, and measure and record the temperature, pH, and specific electrical conductance.
- The fourth and fifth steps will be repeated until groundwater is relatively silt-free; no further change is noted; and the temperature, pH, and specific conductance readings have stabilized to within 10% or 10 well volumes and 5 times the volume of water used to complete the well have been removed.
- The developing equipment will be raised two feet and then Steps 4 and 5 will be repeated.
- Step 6 will be repeated until entire well screen has been developed.

WASTE DISPOSAL

- All waste generated will be disposed in accordance to the methods and procedures contained in the Waste Disposal SOP.
- All water generated during cleaning and development procedures will be collected and contained on site in 55-gallon drums for future analysis and appropriate disposal.
- Personal protective equipment, such as gloves, disposable clothing, and other disposable equipment, resulting from personnel cleaning procedures and from soil sampling and handling activities, will be placed in plastic bags. These bags will be transferred into appropriately labeled 55-gallon drums or a covered roll-off box for appropriate disposal.

EQUIPMENT

- Appropriate health and safety equipment
- Knife
- Power source (generator)
- Field book
- Well Development Form
- Well keys
- Graduated pails
- Pump and tubing
- Cleaning supplies (including non-phosphate soap, buckets, brushes, laboratory-supplied distilled/deionized water, tap water, cleaning solvent, aluminum foil, plastic sheeting, etc.)
- Water level meter
- pH/temperature/conductivity meter
- Clear glass jars (e.g., drillers' jars)

REFERENCE

1. Environmental Protection Agency (1986), RCRA Ground-Water Monitoring Technical Enforcement Guidance Document, OSWER-9950.1.

2. Environmental Protection Agency (1987), A Compendium of Superfund Field Operations Methods, EPA/540/P-87/001.

3. Environmental Protection Agency (1988), Guidance for Conducting Remedial Investigations and Feasibility Studies Under CERCLA, Interim Final, EPA/540/G-89/004.

4.0 AQUIFER CHARACTERIZATION

4.1 MANUAL WATER LEVEL MEASUREMENT PROCEDURE

INTRODUCTION

This procedure describes measurement of water levels in groundwater monitoring and extraction wells, piezometers and boreholes. This procedure does not cover automated measurement of water levels with a transducers/datalogger, and does not cover measurement of phase-separated liquids.

Water levels in monitoring wells will be measured prior to each sampling event and at other times as indicated in the project Work Plan. Water levels will be acquired in a manner that provides accurate data that can be used to calculate vertical and horizontal hydraulic gradients and other hydrogeologic parameters. Accuracy in obtaining the measurements is critical to insure the useability of the data.

PROCEDURES REFERENCED

- 5.3 Non-Aqueous Phase Liquid (NAPL) Monitoring and Sample Collection
- 6.0 Field Instruments – Use And Calibration
- 7.0 Equipment Decontamination

PROCEDURE

In order to provide reliable data, water level monitoring events should be collected over as short a period of time as practical. Barometric pressure can affect groundwater levels and, therefore, observation of significant weather changes during the period of water level measurements must be noted. Tidal fluctuations, navigation controls on rivers, rainfall events and groundwater pumping can also affect groundwater level measurements. Personnel collecting water level data must note if any of these controls are in effect during the groundwater level collection period. Due to possible changes during the groundwater level collection period, it is imperative that the time of data collection at each station be accurately recorded.

In conjunction with groundwater level measurements, surface water (e.g., ponds, lakes, rivers, and lagoons) often are monitored as well. This information is very helpful (and can be critical) in understanding the hydrogeologic setting of the site and most importantly how contaminants may move beneath the site.

The depth to groundwater will be measured with an electronic depth-indicating probe. Prior to obtaining a measurement, a fixed reference point on the well casing will be

established for each well to be measured. Unless otherwise established, the reference point is typically established and marked on the north side of the well casing. Avoid using protective casings or flush-mounted roadboxes for reference, due to the greater potential for damage or settlement.

If provided for in the project Work Plan, the elevation of the reference point shall be obtained by accepted surveying methods, to the nearest 0.01-ft.

The water level probe will be lowered into the well until the meter indicates (via indicator light or tone) the water is reached. The probe will be raised above water level and slowly lowered again until water is indicated. The cable will be held against the side of the inner protective casing at the point designated for water level measurements and a depth reading taken. This procedure will be followed three times or until a consistent value is obtained. The value will be recorded to the nearest 0.01 feet on the Groundwater Level Monitoring Report form or other designated data recording location if specified in the project Work Plan.

Upon completion, the probe will be raised to the surface and together with the amount of cable that entered the well casing, will be decontaminated in accordance with methods described in Equipment Decontamination Procedure.

EQUIPMENT

- Battery-operated, non-stretch electronic water level probe with permanent markings at 0.01 ft. increments (traceable to national measurement standards), such as the Solinst Model 101 or equivalent.

- The calibrated cable on the depth indicator will be checked against a surveyor's steel tape once per quarter year. A new cable will be installed if the cable has changed by more than 0.01% (0.01 feet for a 100-foot cable). See also the Field Instruments – Use and Calibration Procedure.

- Groundwater Level Monitoring Report form.

REFERENCE

- ASTM 4750 Test Method for Determining Subsurface Liquid Levels in a Borehole or Monitoring Well (Observation Well)

- ASTM D6000 Guide for Presentation of Water-Level Information from Ground-Water Sites

4.2 IN-SITU HYDRAULIC CONDUCTIVITY (SLUG TESTING) PROCEDURE

INTRODUCTION

This procedure describes the protocol for performing in-situ hydraulic conductivity (slug) testing, including preparation, collection of valid field data, and preliminary evaluation of the data.

A slug test is performed to assess the hydraulic conductivity of a water-bearing zone. Slug tests are accomplished by stressing the screened water-bearing zone through an instantaneous displacement (with a slug) or removal of water (with a bailer) and subsequently measuring the water level response in the well over time.

Slug testing in select monitoring wells will be performed after the wells have been installed and developed as covered in the RFI Work Plan. Slug testing data will be acquired in a manner that provides valid data that can be used to calculate the horizontal hydraulic conductivity of the formation tested.

There are two types of slug tests: falling-head tests and rising-head tests. It is generally preferable to do a rising-head slug test due to a number of potential problems that can arise with falling-head tests (some of these may lead to inaccurate hydraulic conductivity estimates). Measurements can be collected manually using a battery-operated water level measurement probe or automatically using a datalogger/pressure transducer system.

PROCEDURES REFERENCED

-
- 3.0 Monitoring Wells, Pump Wells and Piezometers
- 4.1 Manual Water Level Measurement
- 6.0 Field Instruments – Use And Calibration
- 7.0 Equipment Decontamination

PROCEDURE

A slug test involves rapidly changing the water level in a well and then measuring the water-level response over time. A very quick change in the water level in a well should be effected at the beginning of a slug test using one of several methods:

- Adding or removing a slug of water
- Inserting or withdrawing a solid or sealed object with an appropriate overall density
- Changing the air pressure in a well (only when a pressure transducer is used)

The method chosen will depend on project needs, equipment availability, water disposal/treatment options, pertinent laws and regulations, and operator experience.

The protocols that follow assume that a person effectively can perform one of the above methods for rapidly changing the water level in a well at the start of a slug test, and can then use either a manual or automatic procedure for measuring water level response over time.

A manual procedure usually is feasible only if a water-bearing zone being tested has a relatively low to moderate hydraulic conductivity (e.g., less than 10^{-3} cm/s). A person can confirm that a manual water-level measurement procedure is feasible using Step 7 below.

If the hydraulic conductivity of a water-bearing zone is relatively high, then water levels in a well may change too rapidly for an accurate manual measurement. In that case, automatic data collection using a pressure transducer and a data logger should be used.

Certain activities should be avoided in slug testing. In general, a person should **not** conduct any type of slug testing in a well if:

- The well contains a pipe or tube of more than nominal diameter, or an obstruction in a depth range where the water level would change
- The casing diameter in a well varies in the depth range where the water level would level change
- The water level in a well has not yet recovered to nearly static conditions (e.g, 95% or more) after a prior disturbance, e.g., drilling, purging, development, previous well testing, etc.
- Non-aqueous phase liquid (NAPL) is present in a well

A ***rising-head*** test should generally **not** be conducted by:

- Bailing multiple times, rather than creating an instantaneous water-level change

- Pumping to remove water, unless the amount of water to be removed by the pump can be removed nearly instantaneously and any back-flush can be eliminated
- Using a bailer that has a leaky check valve
- Using a bailer with a diameter so close to that of the casing that groundwater is suctioned into the well while the bailer is raised

A ***falling-head*** test should generally **not** be conducted if:

- The static water level (SWL) in a well is within the open/screened interval (unsaturated flow)
- Water to be added could be contaminated
- Water to be added contains significant clay, silt, bacteria, algae or other agents that can cause plugging of soil pores, well screen, or rock fractures
- Water to be added is distilled or deionized and could thus mobilize fine particles, leading to plugging and reduced apparent hydraulic conductivity
- The amount of water to be added cannot be added nearly instantaneously (e.g., if pouring takes over 5% of the "90%-recovery" time)
- Laws or regulations prohibit addition of water to the water-bearing zone to be tested

The following data should be obtained prior to heading into the field and/or in the field during slug testing and recorded appropriately (e.g., on appropriate form in a field book, and/or onto an electronic form copied to computer disk):

- Client name
- Site name
- Testing company
- Name of tester
- Date and time of test
- Well number
- Well location
- Well casing diameter
- Well screen or open-hole section diameter
- Total depth of well
- Any unusual well, weather, or hydrologic features or conditions
- Top-of-riser distance above ground surface
- Test procedure used
- Transport and disposal methods for any water removed
- Decontamination procedures
- Problems and solutions to problems encountered during testing
- Static water level

Other information needed for proper slug-test data interpretation includes:

- Depth-interval of screen or open section in well
- Sandpack porosity (if water levels intersect screen)
- Sandpack diameter (if water levels intersect screen)
- Stratigraphic horizon materials and elevations
- Hydraulic conductivity of bounding low-permeability units, if present
- Ground-surface elevation

Manual Data Measurement for "Slow-Recovery" Wells

The steps for conducting a slug test using manual water-level measurements are as follows:

1. Conduct a review of the RFI Work Plan and the Health and Safety Plan with the Project Coordinator, and plan for notifications to responsible parties and for site access, as needed.

2. Gather and inspect equipment needed for operation.

3. Decontaminate all necessary equipment before entering a site and between each slug test. In some instances, but *only* if proper authorization is obtained, equipment may not have to be decontaminated between wells. In those instances, slug tests will be performed starting with the cleanest wells and continuing to more contaminated wells. This, however, requires prior knowledge of contaminant distribution in groundwater, knowledge that is not always available with an acceptable degree of certainty. In all cases, equipment should be decontaminated after the end of the testing program.

4. Measure and record the SWL of the well to be tested.

5. Perform the following pre-test activity if a ***rising-head*** test is to be done:

- Lower the water level probe and record the depth to the base of the well.

- Allow the bailer or slug to slowly move down into the groundwater. If possible, fully immerse the bailer or slug. If there is not enough water in the well for the bailer to be fully immersed, then let the bottom of the bailer gently come to rest on the well bottom, or a few inches above the well bottom. Reduce agitation of the sediment on the bottom of the well as sediment in the bailer may keep the check valve from properly sealing.
- Measure the slowly falling water levels using the probe until the water level in the well re-equilibrates to static conditions (within 0.02 feet of SWL).
- Set the probe below the anticipated depth of the base of the bailer or slug (the most effective position to allow immediate subsequent readings).

6. Start the slug test by creating a nearly instantaneous displacement in water level:

For a ***rising-head*** test:

- Pull the bailer or slug rapidly upward, and either take it out of the well or secure it so that its base always stays above the static water level.
- Immediately start measuring and recording rising water levels in the well at the predetermined time frequency using the probe. Record as quickly as can accurately be done.
- If a bailer is used, listen for cascading water while the bailer is being raised or remains suspended in the well, a sign of check-valve failure; if failure occurs, clean and repair the valve and start over.
- If a bailer is used, measure the volume of water removed by the bailer after retrieval.

For a ***falling-head*** test:

- Use a water level probe to measure the depth to SWL and to the base of the well.
- If a bailer will be used for initial displacement of groundwater, fill it with potable water and make sure that it is not leaking.
- Start the test by quickly lowering the bailer or slug downwards into the groundwater so that, if possible, its top lies below the static water level; if not possible, lower the bailer or slug to a point several inches above the base of the well. If the bailer or slug does not remain fully submersed throughout the test, the interpreter of the data needs to know the depth of the top of the bailer or slug to account for the change in effective well radius once the water level falls below the top.
- Alternatively, if a container of water will be poured into the well to cause the initial displacement, enlarge its opening to the size of the diameter of the well so that pouring can occur rapidly. Start the test by rapidly adding water to the well.
- Immediately start measuring and recording falling water levels over time in the well using the probe. Record levels as quickly as can accurately be done.

7. Continue measuring the water levels as they change over time until the water in the well rises or falls to the limit specified in the RFI Work Plan (usually 90% recovery or 1 hour, whichever comes first). A minimum of 8 to 10 water level measurements should be made prior to stopping the test, with the last data point being collected preferably near the 90%-recovery point, the 1-hour point, or greater. If recovery is sufficiently rapid that the minimum number of points cannot be measured, then re-execute the test using a transducer/data logger.

8. Compare the volume of groundwater recovered in the bailer, if one was used, with the volume of groundwater estimated to have been removed from the well (V) based on the initial recorded water-level displacement (H) and borehole radius (r), e.g., $V=H\pi r^2$. If, for a rising-head test, the static water level lies within the

screened section of the well, then the sandpack porosity (n) and radius (R) must accounted for also in the volume calculation, e.g., $V=H\pi r^2 + nH\pi(R-r)^2$. A similar comparison can be done if a slug is used. If the volume recovered and the calculated volume do not reasonably correlate, based on site-specific conditions, the test should be performed again.

9. Record all data in a field book and all pertinent data onto appropriate form.

10. Decontaminate all necessary equipment in accordance with methods described in Equipment Decontamination Procedure.

11. Properly containerize and label spent decontamination fluid or groundwater removed from the well. Refer to Waste Characterization Procedure for guidance.

12. Lock all well caps and secure the site as needed.

13. Submit the slug-test data to a scientist or engineer assigned by the Project Manager to interpret the data. The data should be interpreted based on an appropriate model for the known hydrogeologic conditions in the field. Interpretation should be performed using a widely accepted computer-based model, or, alternatively, using an analytical model described in one or more journal articles by the originator of the model.

Any variations from these procedures should first be approved by the Project Coordinator or Project Manager.

<u>Automatic (Transducer/DataLogger) Data Measurement for "Fast-Recovery" Wells</u>

The steps for conducting a slug test using automatic water-level measurements are as follows:

1. Conduct a review of the RFI Work Plan and the Health and Safety Plan with the Project Field Supervisor, and plan, as needed, for notifications to responsible parties and for site access.
2. Gather equipment needed and inspect for operation.

3. Decontaminate all necessary equipment before entering a site and between each slug test. In some instances, but *only* if proper authorization is obtained, equipment may not have to be decontaminated between wells. In those instances, slug tests will be performed starting with the cleanest wells and continuing with more contaminated wells. This, however, requires prior knowledge of contaminant distribution in groundwater, knowledge that is not always available with an acceptable degree of certainty. In all cases, equipment should be decontaminated after the end of the testing program.

4. Measure and record the SWL of the well to be tested.

5. Test the pressure transducer and data logger, and obtain well-bottom and SWL pressures, using the following steps:

- Place the pressure transducer at least several feet below the top of water as well as below the projected depth of the lowest part of the bailer or slug to be used.
- Make pressure readings until three uniform values are read consecutively.
- Raise the datalogger one foot from its original position. View the pressure reading to confirm that the transducer accurately reported the change in position.
- Lower the pressure transducer to the base of the well, and measure and record the pressure there.
- Return the transducer to its original position and secure the suspension cable to the well casing. Again, make pressure readings until three uniform values are read consecutively. Compare with the original readings to make sure no drift occurs.

6. Perform the following pre-test activities if a rising-head test is to be performed:

- Allow the bailer or slug that will be used to move down into the groundwater. If possible, fully immerse the bailer. If there is not enough water in the well for the bailer to be fully immersed, then let the bottom of the bailer gently come to rest on the well bottom, or a few inches above the well bottom. Prevent agitation of sediment on the bottom of the well as sediment in the bailer may keep the check valve from properly sealing.
- Measure falling pressures during recovery using the pressure transducer until the water level in the well re-equilibrates to near-static conditions (within 0.02 feet).
- Set the pressure transducer below the base of the immersed bailer or slug.

7. Start the slug test by creating a nearly instantaneous displacement in water level:

For a ***rising-head*** test:

- Pull the bailer or slug rapidly upwards, either removing it from the well or secure/suspend it within the well several feet above the SWL.
- Simultaneously pull bailer and initiate the datalogger, beginning the measuring/recording of rising water levels in the well at the predetermined time frequencies (a logarithmic time scale is usually employed).
- If a bailer is used, listen for cascading water while the bailer is being raised or is suspended, a sign of check-valve failure; if failure occurs, clean and repair the valve and start over.
- If a bailer is used, measure the volume of water removed by the bailer after retrieval.

For a ***falling-head*** test:

- Measure the depth to SWL and to the base of the well using a pressure transducer and a battery-operated water level probe.
- If a bailer is to be used for initial displacement of groundwater, fill it with potable water and make sure it is not leaking.
- Start the test by quickly lowering the bailer (or slug) downwards into the groundwater so that, if possible, its top lies below the static water level; if not possible, lower the bailer (or slug) to a point several inches above the base of the well. If the bailer or slug does not remain fully submersed throughout the test, the interpreter of the data needs to know the depth of the top of the bailer or slug to account for the change in effective well radius once the water level falls below the top.
- Alternatively, if a container of water will be poured into the well to cause the initial displacement, enlarge its opening to the size of the diameter of the well so that pouring can occur rapidly. Start the test by rapidly adding water to the well.
- Immediately after all water has reached the depth of the SWL start measuring and recording falling water levels at the predetermined time frequency in the well using the pressure transducer and data logger (a logarithmic time scale is usually employed)

8. Continue measuring the water levels as they change over time until the water in the well rises or falls to the limit specified in the RFI Work Plan (usually 90% recovery or one hour, whichever comes first). A pre-set logarithmic sampling interval, with increasing intervals of time, is ideal, usually predetermined by the datalogger's default setup.

9. Compare the volume of groundwater recovered in the bailer, if one is used, with the volume of groundwater estimated to have been removed from the well (V) based on the initial recorded water-level displacement (H) and borehole radius (r), e.g., $V=H\pi r^2$. If, for a rising-head test, the static water level lies within the screened section of the well, then the sandpack porosity (n) and radius (R) must accounted for also in the volume calculation, e.g., $V=H\pi r^2 + nH\pi(R-r)^2$. A similar comparison can

be performed if a slug is used. If the volume recovered and the calculated volume do not reasonably correlate, based on site-specific conditions, the test should be performed again.

10. Record all data in a field book and all pertinent data on appropriate form.

11. Decontaminate all necessary equipment in accordance with methods described in Equipment Decontamination Procedure.

12. Properly containerize and label spent decontamination fluid or groundwater removed from the well. Refer to Waste Characterization Procedure for guidance.

13. Lock all well caps and secure the site as needed.

14. Submit the slug-test data to the Project Manager for interpretation. The data should be interpreted based on an appropriate model for the known hydrogeologic conditions in the field. Interpretation should be performed using an accepted, published computer-based model.

Any variations from these procedures should first be approved by the Project Coordinator or Project Manager.

EQUIPMENT

- A battery-operated water level measurement probe, marked in 0.01-foot increments
- Form 5.2-01
- Data logger and laptop computer with fully charged battery (if required)
- A clean bailer or a solid or sealed slug
- Clean rope or string for raising and lowering a bailer or slug
- Appropriate container for withdrawn groundwater and/or decontamination fluids
- If snow or soil removal from over a well might be required, a shovel
- Site-access and well-cap keys, as needed
- Site maps (property lines, wells, topography, etc.), as needed
- If a well to be slug tested is an artesian flowing well, duct tape, couplings, and extra casing of appropriate diameter for increasing casing height so as to enable measurement of a static water level
- Pressure transducer of appropriate pressure range for the depths of water to be tested, if needed
- Appropriate field forms

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5.0 SAMPLE COLLECTION FOR LABORATORY ANALYSIS

5.1 SOIL SAMPLE COLLECTION FOR LABORATORY ANALYSIS

INTRODUCTION

The following procedure describes typical soil sample collection methods for submission of samples to a laboratory for chemical analysis. Two sample situations are presented: soil sampling from surficial soils and soil sampling from sub-surface samplers such as a split-spoon sampler or a direct push sampler.

Soil sampling procedures may vary from project to project due to different parameters of concern, different guidance provided by the state/province where the site is located, or the specific objectives for the project. Therefore, it is essential that the sampling team members carefully review the RFI Work Plan requirements and the rationale behind the program. The primary goal of soil sampling is to collect representative samples for examination and chemical analysis (if required).

Grab Versus Composite Samples

A grab sample is collected to identify and quantify compounds at a specific location or interval. The sample is comprised of no more than the minimum amount of soil necessary to make up the volume of sample dictated by the required sample analyses.

PROCEDURES REFERENCED

- 2.1 Drilling Techniques/Background Information
- 2.2 Soil Borings
- 2.5 Soil Classification
- 5.4 Sample Handling and Shipping
- 7.0 Equipment Decontamination

SAMPLE COLLECTION

1.0 Surficial Soil Sample Collection

1.1 Sample Strategy -Random, Biased, and Grid-Based Sampling

Unless there is a strong indication of contaminant presence, such as staining, then soil sample locations may be randomly selected from several areas within the site.

If any areas show evidence of contamination, such as staining or vegetative stress, biased samples will be collected from each area to characterize the contamination present in each area. Background and control samples are also biased, since they are collected in locations typical of non-Site-impacted conditions.

When soil sampling investigations involve large areas, a grid-based soil sampling program is used. There is no single grid size that is appropriate for all sites. Common grid sizes are developed on 50-foot and 100-foot centers. It is acceptable to integrate several different grid sizes in a single investigation.

For surficial soil sampling programs, it is also important to consider the presence of structures and drainage pathways that might affect contaminant migration. It is sometimes desirable to select sampling locations in low lying areas which are capable of retaining some surface water flow since these areas could provide samples which are representative of historic site conditions (worst-case scenario if surface water flow was a concern).

1.2 Sample Interval

Surficial soils are generally considered to be soil between ground surface and 6 to 12 inches below ground surface. However, for risk assessment purposes, regulatory authorities often consider soil from ground surface to 2 feet below ground surface to be surficial soil. The exact interval to be considered as surficial soil is often a matter of discussion with the regulatory authorities that review the RFI Work Plan. The sample interval is important to the manner in which the data are ultimately interpreted. Another important factor is the type of soil. If there are different types of soil present at the site, this may have a bearing on the sample interval. For example, it may be important to separately sample a layer of material with high organic carbon content that overlies a layer of fine-grained soil.

1.3 Surface Sampling Procedure

Soil sampling techniques are dependent upon the sample interval of interest, the type of soil material to be sampled, and the requirements for handling the sample after retrieval.

The most common method for collection of surficial soil samples involves the use of a stainless steel trowel. Soil samples may also be collected with spoons and push tubes. The sampling equipment is cleaned between sample locations. A typical surficial soil sampling protocol is outlined below:

- Surficial soil samples will be collected using a pre-cleaned stainless steel trowel or other appropriate tool. Each sample will consist of soil from the surface to the depth specified within the Work Plan;

- A new pair of disposable gloves will be used at each sample location;

- Any surficial debris (i.e., grass cover, gravel) should be removed from the area where the sample is to be collected using a separate pre-cleaned device. Gravel presents difficulties for the laboratory in terms of sample preparation and is typically not representative of contaminant concentrations in nearby soil.

- A pre-cleaned sampling tool will be used to remove the sample from the layer of exposed soil.

- When only one sample container is required, the collected soil will be placed directly into the clean, pre-labeled sample jar. When more than one sample container requires filling or samples will be split for duplicate analyses; the soils will first be homogenized in a pre-cleaned stainless steel bowl; and then placed into the respective sample containers. It is important that soil samples be mixed as thoroughly as possible to ensure that the sample is as representative as possible of the sample interval. When round bowls are used for sample mixing, mixing is achieved by stirring the material in a circular motion and occasionally turning the material over. Soil samples collected for volatile organic compounds analyses shall <u>not</u> be mixed.

- Samples will be placed on ice or cooler packs in laboratory supplied shipping coolers after collection.

  Exception is noted for the collection of volatile organic compounds (VOC's) which require special sample collection methods. VOC's are collected directly into a sample vial (triplicate volume typically required) without headspace, or collected using an EnCore Sampler™ (triplicate samples collected per manufacturers instructions). Samples for VOC's are typically collected first, without homogenization or extra handling to limit the loss of volatile constituents.

  The VOC sample collection methodology will be identified in the Work Plan, which will dictate the sample method. The methodology for VOC sampling varies from area to area, so carefull review of this issue in advance of the field efforts is required.

2.0 Sub-Surface Sample Collection

Sub-surface soil sample collection is typically performed with the help of a drill unit, direct-push probing unit, or hand-driven/held samplers. Typically a boring is advanced incrementally to permit intermittent or continuous sampling to the required depth of chemical sample collection; or alternatively sampling may be initiated if certain conditions are observed (i.e. chemical presence or volatile presence identified from monitoring). Sample collection criteria and locations, are normally stipulated by the Work Plan.

Any drilling procedure that provides a suitably clean and stable hole before insertion of the sampler and assures that the penetration test or other sampling technique is performed on essentially undisturbed soil is acceptable. The drilling method is to be selected based on the subsurface conditions. Each of the following procedures have proven to be acceptable for specific subsurface conditions:

- Conventional drilling with continuous flight hollow-stem auger method (with inside diameter between 2.2 and 6.5 inches) using split spoon samplers (Standard Penetration Test – SPT) or Shelby tube samplers; or
- direct - push samplers, advanced using a percussion/vibratory hammer (Geoprobe ™ or equivalent); or
- hand – held/driven split spoon sampling equipment or portable hammer and split spoon sampling equipment (final depth will be limited).

Several drilling methods are not acceptable. These include: jetting through an open tube sampler and then sampling when the desired depth is reached; use of continuous flight solid auger equipment below the groundwater table in non-cohesive soils; casing driven below the sampling depth prior to sampling; and advancing a borehole with bottom discharge bits.

The following subsections describe the specific methods for completing split spoon sampling, shelby tube sampling, and direct-push sampling. Section 2.4 describes the soil sampling procedure for chemical analysis, once a soil core is recovered from any of the above sample collection devices.

2.1 Split – Spoon Sampling Method

This method is used to obtain representative samples of subsurface soil materials for sample collection. The test methods described below must be followed to ensure that the soils captured in the split-spoon or shelby tube are relatively undisturbed/representative of the desired soil interval and obtain accurate Standard Penetration Test (SPT) values. The SPT values reflect the sub-surface soils density and is typically measured when performing geo-technical work or environmental borings. This information although not

directly relevant to the collection of chemical samples, is collected because it is beneficail in terms of stratigraphy interpretation and understanding the conditions below grade.

The split barrel sampler, or split spoon, consists of an 18- or 24-inch long, 2-inch outside diameter tube, which comes apart length wise into two halves. Larger spoons are available for use when a larger sample volume is required (4-inch diameter spoons).

Once the borehole is advanced, by an appropriate method (e.g. hollow stem augers), to the target depth and the borehole cleaned of cuttings, representative soil samples are collected in the following manner:

- the split-spoon sampler should be inspected to ensure it is properly cleaned and decontaminated. The driving shoe (tip) should be relatively sharp and free of severe dents and distortions;
- the cleaned split-spoon sampler is attached to the drill rods and lowered into the borehole. Do not allow the sampler to drop onto the soil;
- after the sampler has been lowered to the bottom of the hole, it is given a single blow to seat it and make sure that it is in undisturbed soil. If there still appear to be excessive cuttings in the bottom of the borehole, remove the sampler from the borehole and remove the cuttings; and
- mark the drill rods in three or four successive 6-inch (0.15 m) increments, depending on sampler length, so that the advance of the sampler under the impact of the hammer can be easily observed for each 6-inch (0.15 m) increment.

The sampler is then driven continuously for either 18 or 24 inches (0.45 or 0.60 m) by use of a 140-pound (63.5 kg) hammer. The hammer may be lifted and dropped by either the cathead and rope method, or by using a trip, automatic, or semi-automatic drop system.

The hammer should free-fall a distance of 30 inches (±1 inches) (760 mm, ±25 mm) per blow. Measure the drop at least daily to ensure that the drop is correct. To ensure a free-falling hammer, no more than 2 1/4 turns of the rope may be wound around the cathead (see ASTM D1586-84). The number of blows applied in each 6-inch (0.15 m) increment is counted until one of the following occurs:

- a total of 50 blows have been applied during any one of the 6-inch (0.15 m) increments described above;
- a total of 100 blows have been applied;
- there is no advancement of the sampler during the application of ten successive blows of the hammer (i.e., the spoon is "bouncing" on a stone or bedrock); or
- the sampler has advanced the complete 18 or 24 inches (0.45 or 0.60 m) without the limiting blow counts occurring as described above.

In some cases where the limiting number of blow counts has been exceeded, the field supervisor may direct the driller to attempt to drive the sampler more if collection of a greater sample length is essential.

On the field form, record the number of blows required to drive each 6-inch (0.15 m) increment of penetration. The first 6 inches is considered to be a seating drive. The sum of the number of blows required for the second and third 6 inches (0.15 m) of penetration is termed the "standard penetration resistance" or the "N-value".

Note: If the borehole has sloughed and there is caved material in the bottom, the split spoon may push through this under its own weight, but now the spoon is partially "pre-filled". When the spoon is driven the 18 or 24 inches representing its supposedly empty length, the spoon fills completely before the end of the drive interval. Two problems arise:

1. the top part of the sample is not representative of the in-place soil at that depth; and

2. the SPT value will be artificially higher toward the bottom of the drive interval since the spoon was packed full. These conditions should be noted on the field log.

The sampler is then removed from the borehole and unthreaded from the drill rods. The open shoe (cutting end) and head of the sampler are partially unthreaded by the drill crew and the sampler is transferred to the field supervisors work surface.

The open shoe and head are removed by hand, and the sampler is tapped so that the spoon separates.

Measure and record the length of sample recovered making sure to discount any sloughed material that is present on top of the sample core.

Caution must be used when conducting SPT sampling below the groundwater table, particularly in sand or silt soils. These soils tend to heave or "blow back" up the borehole due to the difference in hydraulic pressures between the inside of the HSA and the undisturbed soil. To equalize the hydraulic pressure, the inside of the HSA must be filled with water. The drilling fluid level within the boring or hollow-stem augers needs to be maintained at or above the in-situ groundwater level at all times during drilling, removal of drill rods, and sampling. Since heave or blow back is not always obvious to the driller, it is essential that the water level in the borehole always be maintained at or above the groundwater level.

Section 2.4 describes the soil sampling procedure for chemical analysis, once a soil core is recovered from a split spoon sampler.

2.2 Thin-Walled (Shelby Tubes) Sample Method

Thin-walled samplers are used to collect relatively undisturbed samples (as compared to split-spoon samples) of soft to stiff clayey soils. Shelby tubes are commonly used. The

shelby tube has an outside diameter of 2 or 3 inches and is 3 feet long. These undisturbed samples are used for certain laboratory tests of structural properties (consolidation, hydraulic conductivity, shear strength) or other tests (such as collection of soils for chemical analysis) that might be influenced by sample disturbance. Procedures for conducting thin-walled tube sampling are provided in ASTM D1587-94, and are briefly described below.

- the soil deposit being sampled must be cohesive in nature, and relatively free of sand, gravel, and cobble materials, as contact with these materials will damage the sampler;

- clean out the borehole to the sampling elevation using whatever method is preferred that will ensure the material to be sampled is not disturbed. If groundwater is encountered, maintain the liquid level in the borehole at or above groundwater level during the sampling operation;

- bottom discharge bits are not permitted. Side discharge bits may be used, with caution. Jetting through an open-tube sampler to clean out the borehole to sampling elevation is not permitted. Remove loose material from the center of a casing or hollow-stem auger as carefully as possible to avoid disturbance of the material to be sampled;

- place the sample tube so that its bottom rests on the bottom of the hole. Advance the sampler into the formation without rotation by a continuous and relatively rapid motion; usually hydraulic pressure is applied to the top of the drill rods;

- determine the length of advance by the resistance and condition of the formation, but the length shall never exceed 5 to 10 diameters of the tube in sands and 10 to 15 diameters of the tube in clays;

- in no case should the length of advance be greater than the sample-tube length minus an allowance for the sampler head and a minimum of 3 inches for cuttings.

- the tube may be rotated to shear the bottom of the sample 2 to 3 minutes after pressing in, and prior to retrieval to ensure the sample does not slide out of the tube. Lift the weight of the rods off of the tube prior to rotating.

- withdraw the sampler from the formation as carefully as possible in order to minimize disturbance of the sample.

On occasion it may be required that extraction of the sample from the tube be conducted in the field for chemical sample collection. The following procedure should be followed.

- a sample extruder, which consists of a clamp arrangement to hold the tube and a hydraulic ram to push the sample through the tube, is usually mounted on the side of

the rig. To prevent cross-contamination, be certain that the extruder is field cleaned between each sample;

- the sample is then extruded into a carrying tray; these are often made from a piece of 4-inch or 6-inch diameter PVC pipe cut lengthwise. Be certain that the carrying tray is field cleaned between each sample. The sample is carried to the workstation to describe the sample, trim the potentially cross-contaminated exterior, and select the area for sample collection (see section 2.4 collection procedure).

- the shelby tube may then be thoroughly field cleaned and decontaminated for reuse. Since they are thin-walled, the tubes are easily damaged, crimped, or otherwise distorted during handling or pushing. The shelby tube should be inspected before use and any which are significantly damaged should be rejected.

Section 2.4 describes the soil sampling procedure for chemical analysis, once a soil core is recovered from a shelby tube sampler.

2.3 Direct- Push Sample Method

The operation of the direct-push samplers (i.e. Macro-Core ^{TM} Soil Sampler or equivalent) consists of "pushing and/or vibrating" the sampler into the subsurface using a direct-push unit (i.e. Geoprobe ^{TM} soil probing machine or equivalent). The sampler is typically a hollow tube with a threaded drive head, and threaded cutting shoe; provided with an internal sleeve (i.e.liner) that the soil sample is captured in.

Once driven to the required depth, the sampler body/soil liner and soil core is removed from the borehole for inspection and sample collection. Once above grade the sampler is opened by the probe operator and the liner removed and cut open (opened with a dual blade cutting tool), to expose the soil for inspection and sampling.

The sampler body and ends are decontaminated and a new liner is inserted and the sampler reassembled for collection of the next interval. The clean sampler is then advanced back down the same hole to collect the next soil sample. The Macro-Core ^{TM} sampler can be used in either the open-tube or closed-point sampling mode. The open-tube is the most commonly used method, typically employed in stable soil conditions when the borehole does not collapse. The closed-point system seals the cutting shoe opening until the sampler is at the next sample interval, this prevents collapsed soil from entering the sampler as it is advanced back down the hole. Once at the sample depth, the closed-point is unthreaded and released from the cutting shoe area, such that it rides on top of the soil core as it is being driven into the next interval.

Section 2.4 describes the soil sampling procedure for chemical analysis, once a soil core is recovered from a direct-push sampler.

2.4 Soil Core Chemical Sample Collection Procedure

The following describes the collection of soil samples for chemical analysis from a split spoon soil core, shelby tube soil core, or direct-push sample core.

- record soil core recovery and soil stratigraphy data;
- discard upper and lower ends of sample core (3 inches ±);
- if clayey soils are present use a pre-cleaned stainless steel knife to cut the remaining core longitudinally, alternatively if sandy soils are present, use a clean stainless steel spoon to scrape away the soil surface;
- screen the exposed soil surface with a PID to monitor for the presence of volatile organics;
- with a sample knife or spoon, remove soil from the center portion of the core and place in the sample jar (when only one aliquot is required), or
- when more than one aliquot is required place soils in a pre-cleaned stainless steel bowl for homogenization;
- do not sample large stones and natural vegetative debris;
- homogenize the soil and place directly into the sample jars
- properly label sample container; and
- place collected samples on ice or cooler packs in laboratory supplied shipping coolers.

When only one sample container is required, the collected soil will be placed directly into the clean, pre-labeled sample jar. When more than one sample container requires filling or samples will be split for duplicate analyses; the soils will first be homogenized in a pre-cleaned stainless steel bowl; and then placed into the respective sample containers. It is important that soil samples be mixed as thoroughly as possible to ensure that the sample is as representative as possible of the sample interval. When round bowls are used for sample mixing, mixing is achieved by stirring the material in a circular motion and occasionally turning the material over. Soil samples collected for volatile organic compounds analyses shall <u>not</u> be mixed.

Exception is noted for the collection of volatile organic compounds (VOC's) which require special sample collection methods. VOC's are collected directly into a sample vial (triplicate volume typically required) without headspace, or collected in triplicate using an EnCore Sampler™ (triplicate samples collected per manufacturers instructions). Samples for VOC's are typically collected first, without homogenization or extra handling to limit the loss of volatile constituents.
The VOC sample collection methodology will be identified in the Work Plan, which will dictate the sample method. The methodology for VOC sampling varies from area to area, so carefull review of this issue in advance of the field efforts is required.

FIELD NOTES

All conditions at the time of sample collection should be properly documented in the field log book. This should include a thorough description of the collection method, sample characteristics, including grain size, color, and general appearance, as well as date/time of sampling and labeling information. The location of the sampling point should be described in a sketch and three measurements (swing ties) should be taken to adjacent permanent structures so that the sample location can be readily identified in the field at a future date if necessary. It is often advisable to have a licensed land surveyor accurately survey the locations.

DECONTAMINATION

In all sampling scenarios measures to prevent cross-contamination must be employed. The sampling device selected must be constructed of an inert material with smooth surfaces that can be readily cleaned.

Heavy equipment used for test pit operations must also be cleaned between each location when collecting samples for chemical analysis.

EQUIPMENT

- Drilling equipment and soil sampling tools
- Decontamination fluids and rinse water
- Subsurface Boring Log
- Tape Measure
- Water Level Probe
- Appropriate sampling containers

REFERENCE

1. American Society for Testing and Materials (1991), Standard D1452-80, "Practice for Soil Investigation and Sampling by Auger Borings", Annual Book of ASTM Standard, Section 4, Volume 04.08.

2. Environmental Protection Agency (1986), RCRA Ground-Water Monitoring Technical Enforcement Guidance Document, OSWER-9950.1.

3. Environmental Protection Agency (1987), A Compendium of Superfund Field Operations Methods, EPA/540/P-87/001.

4. ASTM D1452-80 Practice for Soil Investigation and Sampling by Auger Borings.

5. ASTM D1586-84 Test Method for Penetration Test and Split-Barrel Sampling of Soils

6. ASTM D1587-94 Practice for Thin Walled Tube Geotechnical Sampling of Soils

7. ASTM D2488-93 Practice for Description and Identification of Soils (Visual-Manual Procedure)

8. ASTM D4700-91 Guide for Soil Sampling from the Vadose Zone

5.2 GROUNDWATER SAMPLE COLLECTION FOR LABORATORY ANALYSIS

INTRODUCTION

This procedure is for the collection of groundwater samples for laboratory analysis.

The following describes two techniques for groundwater sampling: "Low Stress/Low Flow Methods" and "Typical Sample Methods."

"Low Stress/Low Flow" methods will be employed when it is critical to collect groundwater samples where sediment/colloid presence is significant, particularly in fine-grained formations. Analyses typically sensitive to turbidity/sediment issues are Polychlorinated biphenyls (PCBs), Semi-volatile compounds (SVOCs) and metals.

The "Typical Sample Methods" will be employed where the collection of parameters less sensitive to turbidity/sediment issues are being collected (volatile organics - VOCs, and general chemistry).

NOTE: If Non-aqueous phase liquids (NAPL) (light or dense) are detected in a monitoring well, groundwater sample collection will not be conducted and the Project Manager must be contacted to determine a course of action.

PROCEDURES REFERENCED

- 4.1 Manual Water Level Measurement
- 7.0 Equipment Decontamination

PREPARATORY REQUIREMENTS

- Verify well identification and location using borehole log details and location layout figures. Note the condition of the well and inform the Project Coordinator of any necessary repair work required.
- Prior to opening the well cap, measure the breathing space above the well casing with a PID to establish baseline levels. Repeat this measurement once the well cap is opened. If either of these measurements exceeds the air quality criteria in the health and safety plan, field personnel should adjust their PPE accordingly.

- Prior to commencing the groundwater purging/sampling tasks, a water level must be obtained to determine the well volume for hydraulic purposes. Refer to the Manual Water Level Measurement Procedure for details. In some settings it maybe necessary to allow the water level time to equilibrate. This condition exists if a watertight seal exists at the well cap and the water level has fluctuated above the top of screen; creating a vacuum or pressurized area in this air space. Three water level checks will verify static water level conditions or changing conditions.
- Calculate the water volume in the well. Typically overburden well volumes consider only the quantity of water standing in the well screen and riser; bedrock well volumes are calculated on the quantity of water within the open corehole and within the overburden casing.
- Estimate natural groundwater flow rate into well to determine the approximate pumping rate for purging/sampling activities.

WELL PURGING AND STABILIZATION MONITORING (LOW STRESS/LOW FLOW METHOD)

- The preferred method for groundwater sampling will be the low stress/low flow method described below.
- Bladder pumps/submersible variable rate pumps (i.e., Grundfos™ Rediflo or equivalent) are typically employed.
- Slowly lower the pump, safety cable, tubing and electrical lines into the well to the depth specified by the project requirements. The pump intake must be at the mid-point of the well screen to prevent disturbance and resuspension of any sediment in the screen base.
- Before starting the pump, measure the water level again with the pump in the well leaving the water level measuring device in the well when completed.
- Purge the well at 100 to a maximum of 500 milliliters per minute (ml/min). During purging, the water level should be monitored approximately every 5 minutes, or as appropriate. A steady flow rate should be maintained that results in drawdown of 0.3 feet or less. The rate of pumping should not exceed the natural flow rate conditions of the well being sampled. Care should be taken to maintain pump suction and to avoid entrainment of air in the tubing. Record adjustments made to the pumping rates and water levels immediately after each adjustment.
- During the purging of the well, monitor and record the field indicator parameters (pH, temperature, conductivity, oxidation-reduction (redox) reaction potential (ORP), dissolved oxygen (DO), and turbidity) approximately every five minutes. Stabilization is considered to be achieved when the final groundwater flow rate is achieved, and three consecutive readings for each parameters are within the following limits:
  - pH ±0.1 pH units of the average value of the three readings;
  - temperature ±3 percent of the average value of the three readings;

- conductivity ±0.005 milliSiemen per centimeter (mS/cm) of the average value of the three readings for conductivity <1 mS/cm and ±0.01 mS/cm of the average value of the three readings for conductivity >1 mS/cm;
- ORP ±10 millivolts (mV) of the average value of the three readings;
- DO±10 percent of the average value of the three readings; and
- turbidity ±10 percent of the average value of the three readings, or a final value of less than 5 nephelometric turbidity units (NTU).

- Should stabilization not be achieved for all field parameters, purging is continued until a maximum of 20 well screen volumes have been purged from the well. Since low-flow purging (LFP) likely will not draw groundwater from a significant distance above or below the pump intake, the screen volume is based upon a 5-foot (1.4 m) screen length. After purging 20 well screen volumes, purging is continued if the purge water remains visually turbid and appears to be clearing, or if stabilization parameters are varying slightly outside of the stabilization criteria listed above and appear to be approaching stabilization.
- If low-turbidity samples are critical to the project goals, purging will be extended until turbidity has been reduced to 5 NTU or less.
- The pump must not be removed from the well between purging and sampling.

WELL PURGING AND STABILIZATION MONITORING (TYPICAL METHOD)

- Typically peristaltic pumps or bladder pumps or submersible pumps are preferred. In most cases bailer use is not desirable.
- Pump placement is typically performed at the mid-point of the screen.
- Purge the well until three consecutive well volume measurements of temperature and specific conductivity are approximately plus or minus 10 percent and if the pH values are within 1 pH unit of the last three value averages, and the groundwater turbidity values are less than 5 NTU. If stabilization has not occurred within the first five well volumes removed, continue purging and monitoring until eight well volumes have been pumped.
- Groundwater turbidity may be evaluated by a visual examination for sediment/silt presence or use of a nephlometer. Work Plan specific goals may exist for turbidity values that may require extending the purging, or require an alternate pumping system.
- Monitoring well purging is accomplished by using in-place pumps or by a peristaltic, bladder or other appropriate pump, depending on the well depth. The pump/hose assembly or bailer used for purging should be lowered into the top of the standing water column and not deep into the column. Typically pump placement at the mid-point of the screen is adequate.

SAMPLING TECHNIQUES

- If an alternate pump is utilized, the first pump discharge volumes should be discarded to allow the equipment a period of acclimation to the groundwater.
- Samples are typically collected directly from the pump with the groundwater being discharged directly into the appropriate sample container. Avoid handling the interior of the bottle or bottle cap and don new gloves for each well sampled to avoid contamination of the sample.
- Order of sample collection:
  - Volatile organic compounds
  - Semi-volatile organic compounds
  - Total organic carbon (TOC)
  - Total organic halogens (TOX)
  - Extractable organics
  - Total metals
  - Dissolved metals
  - Phenols
  - Cyanide
  - Sulfate and chloride
  - Nitrate and ammonia
  - Radionuclides
- For low stress/low flow sampling, samples should be collected at a flow rate between 100 and 500 mL/min and such that drawdown of the water level within the well does not exceed the maximum allowable drawdown of 0.3 feet.
- The pumping rate used to collect a sample for VOCs should not exceed 100 mL/min. Samples should be transferred directly to the final container 40 mL glass vials completely full and topped with a teflon cap. Once capped the vial must be inverted and tapped to check for headspace/air presence (bubbles). If air is present the sample vial will be discarded, and re-collected until free of air.
- Field filtration will be performed if dictated by the project Work Plan.
- Sample labels/sample identification
- All samples must be labeled with:
  - A unique sample number
  - Date and time
  - Parameters to be analyzed
  - Project Reference ID
  - Samplers initials
- Labels should be secured to the bottle and should be written in indelible inks.

EQUIPMENT/MATERIALS

- pH meter, Conductivity meter, Dissolved Oxygen (DO) meter, Oxidation-reduction (redox) reaction potential (ORP) meter, Nephlometer, Temperature guage
- Field filtration units (if required)
- Purging/sampling equipment
  - Peristaltic Pump (not suitable for VOCs[1]/SVOCs or depths >25 feet);
  - Suction Pumps (not suitable for LFP, VOCs/SVOCs, or depths >25 feet);
  - Submersible Pumps (suitable for VOCs/SVOCs only at low flow rates);
  - Air Lift Pumps (not suitable for VOCs/SVOCs);
  - Bladder Pumps (suitable for LFR and VOCs/SVOCs);
  - Inertia Pumps (gaining acceptability for VOCs/SVOCs generally not suited for Delphi programs); and
- Water Level Probe
- Sampling Materials (containers, log book/forms, coolers, chain-of-custody)
- Work Plan
- Health and Safety Plan

NOTE[1]: PERISTALTIC PUMP USE FOR VOC COLLECTION IS NOT ACCEPTABLE ON EPA/RCRA SITES; THIS TECHNIQUE HAS GAINED ACCEPTANCE IN SELECT AREAS WHERE IT IS PERMISSIBLE TO COLLECT VOCS USING A PERISTALTIC PUMP AT A LOW FLOW RATE (EX. MICHIGAN).

FIELD NOTES

- Field notes must document all the events, equipment used, and measurements collected during the sampling activities. SOP 1.2 describes the data/recording procedure for field activities. The log book should document the following for each well sampled:
  - Identification of well
  - Well depth
  - Static water level depth and measurement technique
  - Sounded well depth
  - Presence of immiscible layers and detection/collection method
  - Well yield – high or low
  - Purge volume and pumping rate
  - Time well purged

- Measured field parameters
- Purge/sampling device used
- Well sampling sequence
- Sampling appearance
- Sample odors
- Sample volume
- Types of sample containers and sample identification
- Preservative(s) used
- Parameters requested for analysis
- Field analysis data and method(s)
- Sample distribution and transporter
- Laboratory shipped to
- Chain of custody number for shipment to laboratory
- Field observations on sampling event
- Name collector(s)
- Climatic conditions including air temperature
- Problems encountered and any deviations made from the established sampling protocol.

A standard log form for documentation and reporting groundwater purging and sampling events are presented on Form 6.3-01 (Well Purging Field Information Form), 6.3-02 (Sample Collection Data Sheet), and 6.3-03 (Monitoring Well Record for Low-Flow Purging).

GROUNDWATER/DECON FLUID DISPOSAL

- Groundwater disposal methods will vary on a case-by-case basis but may range from:
  - Off-site treatment at private treatment/disposal facilities or public owned treatment facilities
  - On-site treatment at Facility operated facilities
  - Direct discharge to the surrounding ground surface, allowing groundwater infiltration to the underlying subsurface regime
- Decon fluids should be segregated and collected separately from wash waters/groundwater containers.

REFERENCE

1. ASTM D5474 Guide for selection of Data Elements for Groundwater Investigations
2. ASTM D4696 Guide for pore-liquid sampling from the vadose zone
3. ASTM D5979 Guide for conceptualization and characterization of groundwater systems
4. ASTM D5903 Guide for planning and preparing for a groundwater sampling event
5. ASTM D4448 Standard guide for sampling groundwater wells
6. ASTM D6001 Standard guide for direct push water sampling for geo-environmental investigations.
7. USEPA Low-flow (minimal drawdown) ground-water sampling procedures (EPA/540/S-95/504)
8. USEPA RCRA Groundwater Monitoring: Draft Technical guidance (EPA/530-R-93-001)

5.3 NON-AQUEOUS PHASE LIQUID (NAPL) MONITORING AND SAMPLE COLLECTION

INTRODUCTION

This procedure is for monitoring the presence of dense and light non-aqueous phase liquids (DNAPL and LNAPL), and collection of NAPL samples for laboratory analysis in monitoring, observation, and extraction wells.

It should be noted that groundwater sampling and analysis should not be performed in locations where NAPL has been identified.

PROCEDURES REFERENCED

- 4.1 Manual Water Level Measurement
- 7.0 Equipment Decontamination

NAPL LEVEL MEASUREMENTS

- Conduct well identification, inspection, and opening in accordance with the Manual Water Level Measurement Procedures.
- NAPL level measurements are best conducted using a dual phase interface probe. The interface probe uses an optical liquid sensor, in conjunction with an electric circuit to detect the top of a phase-separated liquid and the interface between the phase layer and water (water level). The procedure for use of this probe is:
- For LNAPL:
  - Lower the probe tip into the center of the well until discontinuous beeping is heard (this indicates the top of the LNAPL has been detected). Grasp the calibrated tape at the reference point and note reading. Confirm the reading by slowly raising and lowering the probe to the level of the phase layer.
  - Once the top of the phase layer is confirmed, slowly lower the probe until a continuous sound is heard. This indicates that the water level has been encountered. Grasp the tape at the reference point and note the reading. Confirm this water level measurement.
  - Decontaminate the submerged end of the tape and probe prior to the next use in accordance with the RFI Work Plan requirements.
- For DNAPL:
  - Lower the probe tip in the center of the well to the bottom of the well, a discontinuous beeping will be heard if DNAPL is present. Grasp the calibrated tape at the reference point and note reading.
  - Once the bottom of the well is confirmed, slowly raise the probe until a continuous sound is heard. This indicates that the water level has been encountered and represents the top of the DNAPL layer. Grasp the tape at the reference point and note the reading. Confirm this water level measurement.
  - Decontaminate the submerged end of the tape and probe prior to the next use.

- Alternative NAPL measurement methods exist in the event an interface probe is unavailable or not functioning properly. These methods tend to be less accurate than the interface probe but may be used to establish an estimated NAPL measurement.
  - Clear Bailer – A clear bottom-loading bailer may be used to estimate NAPL thickness if floating or denser than water. If NAPL presence is suspected, the bailer is carefully lowered to the location of suspected NAPL presence (top of water column/base of water column), and slowly removed and examined for NAPL. If present, the column of NAPL within the clear bailer can be measured to estimate the NAPL thickness within the groundwater column.
  - Weighted Cord – Primarily used for DNAPL measurements, a weighted "cotton" string or cord may be lowered to the base of the well and inspected upon retrieval. Typically, the lower DNAPL layer will "coat" the string indicating the approximate thickness of this layer.

WELL NAPL SAMPLING

- Prior to sampling, the level of NAPL in the well should be measured as identified above.
- Various sampling devices can be employed to acquire fluid samples from the top and bottom of the well, including the following:
  - Bottom-loading bailer;
  - Double check value bailer (produces most reliable results);
  - Peristaltic pump for shallow wells (<25 feet in depth); or
  - Inertia pump for deeper wells (up to 300 feet in depth).
- Transfer NAPL to sample containers for shipment to laboratory. NAPL can be sampled to evaluate the physical properties of the fluid or to evaluate chemical composition.
- Decontaminate equipment prior to next use.

Note: Groundwater sampling shall not be performed in locations where NAPL is present.

EQUIPMENT/MATERIAL

- Interface Probe;
- Bottom-loading bailer;
- Double check valve bailer;
- Peristaltic pump;
- Inertia pump
- RFI Work Plan
- Health and Safety Plan
- Field Log Book

REFERENCE

1. Cohen, Robert M., Mercer, James W. (GeoTrans, Inc.), Robert S. Kerr Environmental Research Laboratory "DNAPL Site Evaluation" Office Research and Development. U.S. Environmental Protection Agency
2. Cohen, R.M., Brayda, A.P., Shaw, S.T., and Spaulding, C.P.; Fall 1992 "Evaluation of Visual Methods to Detect NAPL in Soil and Water", Groundwater Monitoring Review, Vol. 12 No. 4, pp. 132-141.

5.4 SURFACE WATER SAMPLE COLLECTION

INTRODUCTION

The procedures presented in this attachment were developed to provide a uniform, standardized approach to collect representative surface water samples. Surface water as referenced herein refers to standing or flowing water above ground surface.

How a sample is collected and handled can affect the quality and interpretation of the sampling results. These procedures are intended to minimize problems associated with variability in sample collection.

PROCEDURES REFERENCED

- 1.2 Field Data Recording
- 4.1 Manual Water Level Measurement
- 5.6 Sample Handling and Shipping
- 7.0 Equipment Decontamination

SAMPLE COLLECTION

- Sample bottles will be labeled prior to sampling using indelible ink.

- Sample locations and analyses will be selected with respect to specific site conditions in RIF. Based on the selection of sample analyses, appropriate sample containers, preservatives, and holding times are stipulated in the QAPP.

- Cover ground with new plastic/polyethylene sheet at each sample location

- Submerge sample container with cap in place with minimal disturbance to surface with open end pointing up upstream. In situations where using the sample container is not feasible, such as with pre-preserved sample containers, a spare sample container will be used as a transfer vessel, and discarded after use.

    - Samples should be collected from downstream (flow) to upstream (flow), so disturbance from sampling does not affect sample quality at other locations. All samples should be collected before any physical features of the stream are measured and prior to sediment sampling.

- Slowly fill container continuously. Use the cap to regulate water flow.

- Lift container from water with minimal disturbance.

- Preserve and filter (for metals) as necessary.

    - Water samples that will be tested for dissolved metals will be filtered in the field with a 0.45 micron filter or cartridge or according to procedures described in the U.S. EPA SW-846 Manual, Method 3005. The liquid phase will be acidified with nitric acid, for preservation.

    - Sample glassware should be filled to the very top of the container for volatile organic analysis (VOA). Once capped the vial must be inverted and tapped to check for headspace/air presence (bubbles). If air is present the sample vial will be discarded, and re-collected until free of air.

- Ensure presence of Teflon septum/liner and secure cap tightly.

- Place immediately into cooler containing ice along with necessary trip blanks. The samples will be stored in coolers, in which ice will be placed in plastic zip-lock bags to contain ice water. Samples will be adequately layered in ice to prevent breakage of glassware. Samples will be positioned upright in the cooler except in the case of 40 milliliter VOA samples. All 40-milliliter VOA vials will be sealed in thick or heavy-duty plastic zip lock bags. If samples are to be shipped to a laboratory for analysis, a Chain of Custody record, custody seals, fragile markers, and reinforced nylon tape will be affixed to the sample cooler. If samples are to be delivered to the contract lab directly, then only the Chain-of-Custody record is required.

    - <u>Chain-of-Custody Form</u> - enclose in large plastic zip lock bag and either tape to inside top of cooler lid or set on top of vermiculite inside of cooler.

    - <u>Custody Seals</u> - place custody seal over cooler gasket separating the cooler bottom at all sides except hinged side.

    - <u>Nylon Tape</u> - tape completely around cooler at two locations. Tape reinforcing should prevent cooler from opening should lid locking mechanism fail.

    - <u>Fragile Markers</u> - fragile markers and upright stickers should be affixed to each side of the cooler.

- Verify that all required documentation entries have been made to the Chain of Custody.

FIELD NOTES

A number of field measurements and observations should be made in the field at the time of sampling. This information is listed below and will be recorded in a project Field Log Book or Sampling Record Form (Haley & Aldrich Form 3202), whichever is more appropriate. For large sampling programs both documentation reports may be completed. For smaller sampling assignments the Project Manager will identify which reporting format is to be used by field personnel. The following list of measurements and observations represents a minimum requirement for surface water samples:

- Field Log Book Entry for Surface Water Samples

  - Surface Water Location Number
  - Time
  - Date Collected
  - Sample Characteristics (measured in separate transfer container not in sample glassware), if requested by the Project Manager - pH, Conductivity, Dissolved Oxygen, Temperature
  - Preservative(s)
  - Samplers (names of individuals who actually collected samples)
  - Sample Destination (Analytical Laboratory)
  - Description (in detail) of the Sample Location
  - Photograph Number and Roll Used, if applicable
  - Approximate dimensions of Stream or Water Course at Sampling Point (measured after sampling) including depth and width
  - Velocity of Water (estimated from floating object)
  - Flow Rate of Water Course (in cubic feet/second; using the following formula)

    Where -

    Q = discharge in cubic feet/second (cfs)
    A = area in square feet
    u = velocity in feet/second (ft/sec)
    w = width in feet
    d = depth in feet
    $Q = Au = wdu$

    The cross sectional area (A) can then be multiplied by the velocity to derive a rough flow rate. Using a watch, measure the time it takes a floating object to travel a measured distance (two or three feet) to calculate the velocity of the water course.

  - Observable Physical Characteristics - Odor, Color, Turbidity, Immiscible Layers, Formation of Precipitates
  - Evidence of Stressed Vegetation, Wildlife, or Dumping

- Ambient Weather Conditions during Sampling - Air Temperature, Sky Condition, Recent Precipitation or Drought
- Samples Collected (enter all sample numbers collected at this location)

- Chain-of-Custody and Request-for-Analysis Forms

  - All samples submitted to the laboratory will be accompanied by a Chain-of-Custody as described in the QAPP.
  - Appropriate Chain-of-Custody procedures will be followed at all times during a sampling event and subsequent transport to the laboratory.

EQUIPMENT

- Decontamination fluids, rinse water and equipment (brushes, towels)
- Appropriate sampling containers
- pH meter, Conductivity meter, Dissolved Oxygen (DO) meter, Temperature gauge
- Sampling Materials (containers, log book/forms, coolers, chain-of-custody)
- Work Plan
- Health and Safety Plan
- Gloves, polyethylene sheeting

DECONTAMINATION

- Sampling equipment such as spatula, measuring tape and trowel will be cleaned prior to and between each use according to procedures outlined in the QAPP. Transfer containers will be decontaminated before disposal.

REFERENCE

1. Environmental Protection Agency (1991), Compendium of ERT Surface Water and Sediment Sampling Procedures, OSWER-9360.4-03.

5.5 SEDIMENT SAMPLING

INTRODUCTION

The field procedures presented in this attachment were developed for obtaining representative samples of sediment.

Sediment samples as referenced herein mean sediment soils or soil-like material below surface water. These samples may contain contaminants that are insoluble in water, persistent in the environment, relatively immobile in the soil, or have low volatility.

These procedures are intended specifically to minimize problems associated with variability in sample collection.

PROCEDURES REFERENCED

- 1.2 Field Data Recording
- 5.6 Sample Handling and Shipping
- 7.0 Equipment Decontamination

SAMPLE COLLECTION

- Label and number required sample bottles. Fill out the label in indelible ink and carefully and clearly address all categories and parameters.
- Sample analyses will be specified in the RFI. Based on the selection of analyses, appropriate containers are identified in the QAPP.
- Decontaminate sampling device and/or container immediately prior to use according to procedures in the QAPP.
- If surface water samples are to be collected from same area as the sediment samples, the surface water samples should be taken first.
- Samples should be collected from downstream (flow) to upstream (flow) so disturbance from sampling does not affect sample quality at other sample locations.

- Samples will be taken with pre-cleaned hand scoops or trowels constructed of stainless steel. A stainless steel laboratory scoop is a preferred scoop device due to its non-corrosive nature.

    - Procedures for Use

        - Insert scoop or trowel into material and remove sample. Care should be exercised during removal of the sample through the surface water to minimize disturbance and loss of material.

        - Decanting the sample is not necessary. Any water present from saturated sediment is representative of sediment.

        - VOCs should be sampled with ENCORE samplers first, directly off the trowel. The remainder of sample should be homogenized using a stainless steel mixing bowl or Teflon tray and then placed into the appropriate sample bottles with a stainless steel lab spoon, wooden tongue depressor or equivalent.

- Sample glassware will be filled to the top. Refer to the QAPP for sample volume size and appropriate containers for given analyses. No preservatives are required for sediment samples. All container caps will include an inner Teflon septa or lining and must be tightly secured to contain the sample.

- Check for appropriate liner in cap and secure cap tightly. Store the samples with ice in an ice chest, following these sealing and packing procedures:

    - Ice will be placed in plastic zip-lock bags to contain ice water. Samples will be adequately layered in ice to prevent breakage of glassware.

    - Check to make sure all appropriate information is in Field Log Book or Sampling Record Form, Chain-of-Custody form and Request for Analysis form (if applicable) using indelible ink.

    - If samples are to be shipped to a laboratory for analysis, a Chain-of-Custody record, custody seals, fragile markers, and reinforced nylon tape will all be properly affixed to or on the sample cooler. If samples are to be delivered to the lab directly then only the Chain-of-Custody record is required.

        - Chain-of-Custody Form - enclose in large plastic zip lock bag and either tape to inside top of cooler lid or set on top of vermiculite inside of cooler.

- Custody Seals - place custody seal over cooler gasket separating the cooler lid from the cooler bottom at all sides except hinged location.

- Nylon Tape - tape completely around cooler at two locations. Tape reinforcing will prevent cooler from opening if the lid locking mechanism fails.

- Fragile Markers - fragile markers and upright stickers will be affixed to each side of the cooler.

FIELD NOTES

This information is listed below and will be recorded in a project Field Log Book or Sampling Record Form, whichever is more appropriate. For large sampling programs both documentation reports will be completed. For small sampling assignments the Project Manager will identify which report is to be used. The following list of measurements and observations represent a minimum requirement for sediment/soil samples:

- Field Log Book Entry For Sediment/Wetland Soil Samples

  - Sediment/Soil Location Number
  - Time
  - Date Collected
  - Samplers (names of individuals who actually collected samples)
  - Sample Destination (Analytical Laboratory) to receive samples
  - Description of Sample Location with Sketch or Map (i.e., sludge lagoon, test pit, boring, stream, etc.)
  - Sample Depth (i.e., distance in feet from ground surface)
  - Photograph Number and Roll used (if applicable).
  - Water Body Characteristics (i.e. size, depth, estimated flow rate)
  - Observable Physical Characteristics:
    - Soil Classification (i.e. silty sand, clay)
    - Odor
    - Color
    - Density, Consistency, etc.
    - Layering
    - Other
  - Evidence of Stressed Vegetation or Wild Life in Area where Sample was taken
  - Ambient Weather Conditions during Sampling:
    - Air Temperature

Sky Condition
Recent Precipitation or Drought

- Samples Collected (enter all sample numbers collected at this location)

- Chain-of-Custody and Request-for-Analysis Forms

  - A Chain-of-Custody form, as shown in the QAPP, will accompany all samples submitted to the laboratory.
  - Appropriate Chain-of-Custody procedures will be followed at all times during a sampling event and subsequent transport to the laboratory. These procedures are described in QAPP in Appendix B.

EQUIPMENT

- Stainless Steel or Teflon Scoop or Trowel, no chrome plated or painted
- Decontamination fluids, rinse water and equipment (brushes, towels)
- Sampling Materials (containers, log book/forms, coolers, chain-of-custody)
- Work Plan
- Health and Safety Plan
- Field Notebook or Sampling Forms
- Polyethylene sheeting, gloves, waste/rubbish container

DECONTAMINATION

Soil/sediment sampling equipment brought into actual contact with a laboratory sample, other than sample containers, will be cleaned prior to an between each use according to procedures outlined in the QAPP.

REFERENCE

1. Environmental Protection Agency (1991), Compendium of ERT Surface Water and Sediment Sampling Procedures, OSWER-9360.4-03.

5.6 SAMPLE NAMING, HANDLING, AND SHIPPING

INTRODUCTION

Sample management is the continuous care given to each sample from the point of collection to receipt at the analytical laboratory. Good sample management ensures that samples are properly recorded, properly labeled, and not lost, broken, or exposed to conditions that may affect the sample's integrity.

All sample submissions must be accompanied with a chain-of-custody (COC) document to record sample collection and submission.

The following sections provide the minimum standards for sample management.

PROCEDURES REFERENCED

A) Field Handling

Prior to entering the field area where sampling is to be conducted, especially at sites with defined exclusion zones, the sampler should ensure that all materials necessary to complete the sampling are on hand.

If samples must be maintained at a specified temperature after collection, proper coolers and ice/cool-packs must be brought out to the field. Consideration should be given to keeping reserve cooling media on hand if sampling events will be of long duration. Conversely, when sampling in extremely cold weather, proper protection of water samples, trip blanks, and field blanks must be considered.

Personnel performing groundwater sampling tasks must check the sample preparation and preservation requirements to ensure compliance with the RFI Work Plan, Quality Assurance Project Plan. Typical sample preparation may involve pH adjustment (i.e., preservation), sample filtration and preservation, or simply cooling to 4°C. Sample preparation requirements vary from site to site and vary depending upon the analytical method for which the samples will be analyzed.

The sampling personnel must also confirm before the sample event, the amount of bottle filling required for the respective sample containers. VOC samples must not have any headspace within the sample collection vial; whereas when collecting select analytes (i.e., metals) a headspace must be provided to allow addition of the required preservative.

B) Sample Labeling

Samples must be properly labeled as soon as practical after collection.

Note that the data shown on the sample label is the minimum data required. The sample label data requirements are listed below for clarity.

i) Project name.
ii) Sample number.
iii) Sampler's initials.
iv) Date of sample collection.
v) Time of sample collection.
vi) Analysis required.
vii) Preservatives.

Quite often the analytical laboratory supplying the containers will provide blank sample labels. If these are adequate and convenient they can be used.

Under certain field conditions it is impractical to complete and attach sample labels to the container at the point of sample collection. However, to ensure that samples are not confused, a clear notation should be made on the container with a permanent marker indicating the last three digits of the sample number. If the containers are too soiled or small for marking, the container can be put into a "zip-lock" bag which can then be labeled.

No one sample number format is adequate for every type of sampling activity. Prior to the start of every project or sub-sampling event within the project, Project Managers and field personnel should devise a sample number format. Sample number formats should be as simple and short as possible. Sample number formats will reduce transcription errors by both Consultants and lab personnel. The sample number format should be comprehensive enough to allow for easy location of detailed sample data within the Site log books. Sample format must also be consistent with any future data management activities.

Unless otherwise instructed, labels should not contain specific names of the sample source (i.e., "Well No. 16"). Provision of such specific data on the label can produce biased lab results.

Sample Labels/Sample Identification

All samples must be labeled with:

- a unique sample number (never to be re-used, nor likely to be);
- date and time;
- parameters to be analyzed; and

- sampler's initials.

Labels should be secured to the bottle and should be written in indelible ink. It is also desirable to place wide clear tape over the label before packing in a cooler for label protection during transportation.

The unique sample identification number may follow the format recommended below, or a specific sample protocol for labeling may be specified in the project RFI Work Plan.

XXXX-XXXXXX-XXXX

This format has been selected to maximize the information content of the sample number. Minor modifications are certainly reasonable.

i) XXXX – Employee four-digit identification number
ii) XXXXXX – Sample Date – Month/Date/Year
iii) XXXX – Time (military)

Note that for Field Duplicates, Field Blanks, Equipment Blanks, Trip Blanks, Field Replicate, Known Duplicate, and Material Blank time will NOT be used. Instead we will use a sample number (ie. 0001, 0002, 0003), reset every day. This will simplify sample naming for the QA/QC samples and avoid identifying the parent sample for blind duplicates.

The decision of how to assign sample numbers will be made at the beginning of a job or phase, and should be consistent throughout the job.

C) Packaging

When possible, sample container preparation and packing for shipment should be completed in a well-organized and clean area, free of any potential cross-contaminants.

Sample containers should be prepared for shipment as follows:

i) Containers should be wiped clean of all debris/water using paper towels (paper towels must be disposed of with other contaminated materials).
ii) Clear, wide packing tape should be placed over the sample label for protection.

While there is no one "best" way to pack samples for shipment, the following packing guidelines should be followed.

i) Plan time to pack your samples (and make delivery to shipper if applicable). Proper packing and manifesting takes time. A day's worth of sampling can be easily wasted due to a few minutes of neglect when packing the samples.
ii) Always opt for more coolers and more padding rather than crowd samples. The cost associated with the packing and shipment of additional coolers is usually

always small in comparison with the cost of having to re-sample due to breakage during shipment.

iii) Do not bulk pack. Each sample must be individually padded.

iv) Large glass containers (1 liter and up) require much more space between containers.

v) Ice is not a packing material due to the reduction in volume when it melts.

The following is a list of standard guidelines that must be followed when packing samples for shipment.

i) When using ice for a cooling media, always double bag the ice in "Zip-Lock" bags.

ii) Double-check to ensure trip and temperature blanks have been included for all shipments containing VOCs, or where otherwise specified in the QA/QC plan.

iii) Enclose the Chain-of-Custody form in a "Zip-Lock" bag.

iv) Ensure custody seals (two, minimum) are placed on each cooler. Coolers with hinged lids should have both seals placed on the opening edge of the lid. Coolers with "free" lids should have seals placed on opposite diagonal corners of the lid. Place clear tape over custody seals.

v) Ensure that all "Hazardous Material" stickers/markings have been removed from coolers being used which previously contained such materials.

Note: Never store sterile sample containers in enclosures containing equipment which use any form of fuel or volatile petroleum based product. An alternate means of secure storage must be planned for.
When conducting sampling in freezing conditions at sites without a heated storage area (free of potential cross contaminants), trip blanks not being used in a QA/QC role should be isolated from coolers immediately after receipt. Trip blanks should be double-bagged and kept from freezing.

D) Chain-of-Custody Records

Chain-of-custody forms will be completed for all samples collected. The form documents the transfer of sample containers.

The chain-of-custody record, completed at the time of sampling, will contain, but not be limited to, the sample number, date and time of sampling, and the name of the sampler. The chain-of-custody document will be signed and dated by the sampler when transferring the samples.

Each sample cooler being shipped to the laboratory will contain a chain-of-custody form. The chain-of-custody form will consist of four copies which will be distributed as follows: The shipper will maintain a copy while the other three copies will be enclosed in a waterproof envelop within the cooler with the samples. The cooler will then be sealed

properly for shipment. The laboratory, upon receiving the samples, will complete the three remaining copies. The laboratory will maintain one copy for their records. One copy will be returned to the Field Coordinator upon receipt of the samples by the laboratory. One copy will be returned with the data deliverables package.

Chain-of-custody (COC) records are legal documents. They must be completed and handled accordingly.

The following list provides guidance for the completion and handling of all COCs.

i) COCs used should be Consultant-standard forms or those supplied by the analytical laboratory. Do not use any COC forms from other labs, even if the heading is blocked out.
ii) COCs must be completed in black ballpoint ink only.
iii) COCs must be completed neatly using printed text.
iv) If a simple mistake is made, line out the error with a single line and initial and date next to it.
v) Each separate sample entry must be sequentially numbered.
vi) The use of "Ditto" or quotation marks to indicate repetitive information in columnar entries should be avoided. If numerous repetitive entries must be made in the same column, place a continuous vertical arrow between the first entry and the next different entry.
vii) When more than one COC form is used for a single shipment, each form must be consecutively numbered using the "Page ___ of ___" format.
viii) If necessary, place additional instructions directly onto the COC. Do not enclose separate loose instructions.
ix) Include a contact name and phone number on the COC in case there is a problem with the shipment.
x) Do not indicate the source of the sample as this may produce a biased lab result.
xi) Before using an acronym on a COC, define clearly the full interpretation of your designation [i.e., Polychlorinated Biphenyls - (PCBs)].

E) <u>Shipment</u>

In all but a few cases the QA/QC plan for the field work will require shipment of samples by overnight carrier. A great many problems can be avoided by proper advance planning. Prior to the start of the field sampling, the carrier should be contacted to determine if pickup can be made at the field site location. If pickup at the field site can be made, the "no-later-than" time for having the shipment ready must be determined.

If no pick-up is available at the Site, the nearest pick-up or drop-off location should be determined. Again, the "no-later-than" time for each location should be determined.

Sample shipments must not be left at unsecured or questionable drop locations (i.e., if the cooler will not fit in a remote drop box do not leave the cooler unattended next to the

drop box). Some overnight carriers do not in fact provide "overnight" shipment to/from some locations. Do not assume; call the carrier in advance before the start of the field work.

Copies of all shipment manifests must be maintained in the field file.

5.7 Vertical Water Quality Profiling

INTRODUCTION

Environmental investigations often involve characterizing the presence of contaminant compounds in groundwater. Such characterization is accomplished a number of ways depending on the site conditions, project objectives and regulatory requirements. Traditionally, site groundwater characterization has involved the installation of monitoring wells for sampling. In recent years, more refined methods of groundwater characterization have been developed. Several studies have shown that vertical mixing of contaminants in groundwater is limited, and that contaminant concentrations can vary by several orders of magnitude within very short vertical distances, even less than one ft. As such, installation of conventional monitoring wells, which typically have screen lengths of 5 to 10 feet or more, cannot discern the presence of narrow zones of significant contamination. Moreover, high concentrations of contaminant compounds in a narrow zone can be significantly diluted by cleaner-zone water entering into the well screen from outside the high concentration zone.

As traditional rotary drilling and sampling methods have been supplemented by direct-push methods, equipment has been developed that facilitates sampling groundwater from small, discrete sampling intervals. By obtaining a series of samples in a borehole, a vertical water quality profile can be developed. Such profiling can provide a more detailed understanding of site contaminant distribution and migration than might otherwise have been practical using only standard monitoring well methodology. It can be especially helpful for sites with variable soils that contain seams or zones of greater permeability where contaminants migrate more readily. Depending on regulatory or work plan requirements, the discrete, in-situ samples may need to be combined with standard monitoring well samples.

Vertical water quality profiling is most often performed in overburden soils, however discrete groundwater sampling can also be performed in bedrock, albeit using more time-consuming and costly methods.

Although this SOP cannot include a comprehensive discussion of all available methods and equipment, it presents general guidelines for vertical water quality profiling.

PROCEDURES REFERENCED

- 2.2 Drilling Techniques
- 2.3 Soil Borings
- 2.4 Bedrock Coring
- 2.5 Borehole Abandonment and Sealing
- 5.4 Water Pressure Testing
- 6.3 Groundwater Sampling
- 9.0 Equipment Decontamination

PROCEDURES AND GUIDELINES

In order to obtain a vertical profile of water quality, several discrete samples must be obtained at a subsurface exploration location. A discrete sample contains groundwater from within a short vertical section of the formation being tested, usually less than one foot. It must be obtained in a manner that does not allow the sample to be affected by groundwater from above or below the target interval, by drilling or other fluid in the borehole or casing, or by residual fluid in the sampler from previous samples. Thus procedures and sampling equipment designed with these restrictions in mind must be used.

A variation on the method of actually obtaining a sample of groundwater involves advancing through the soil a probe containing a detector designed to continuously measure levels of volatile organic compounds (VOCs). This method was developed by Geoprobe™ and utilizes their Membrane Interface Probe (MIP). This is discussed in further detail below.

Overburden

Several products have been developed to obtain discrete groundwater samples in overburden soils. These include, but are not limited to:

- Hydropunch™, by QED
- Groundwater Profiler, by Geoprobe™
- Screenpoint 15, by Geoprobe™

- Waterloo Profiler, by the University of Waterloo, Ontario

Each of these tools is generally deployed using direct-push methods, although conventional drop hammer methods can also be used. The tools are advanced by driving them down through saturated overburden soils. Once the desired depth of sampling is reached, a sample of groundwater is obtained from a narrow depth range.

Sample collection is performed differently manner depending on the type of tool used. For the Hydropunch and Screenpoint 15, the outer driving rods are retracted after reaching the desired depth, exposing an inner screened portion of the sampler that allows entry of groundwater into the sample chamber. At that point a sample of the groundwater is withdrawn from the sampling chamber and up through the drill rods using any of the following:

  - a foot valve (Waterra or similar product) and tubing,
  - a peristaltic pump connected to tubing,
  - a small diameter bailer,
  - or other methods.

For the Groundwater Profiler, 6-inch or 12-inch sections of exposed screen are driven to the desired depth. Deionized water can be circulated down the drill rods, through the screen, and into the formation during advancement to prevent screen clogging or entry of unwanted groundwater from non-target zones. A sample is then obtained using one of the sample methods described above.

For the Waterloo Sampler, sample withdrawal tubing is connected to small screened ports in the probe. Deionized water is pumped downward through the tubing and into the formation during probe advancement to minimize clogging of the sampling ports. The tubing passes through the inside of the drive rods to the surface. Once the desired sampling depth is achieved, the tubing is connected to a peristaltic pump at the surface that extracts a groundwater sample.

In the case of the Groundwater Profiler and Waterloo Sampler, the tool is designed to remain in the borehole throughout the profiling effort. In others, the probe and screen may require removal and decontamination after each sample. SOP 9.0, Equipment Decontamination, should be referenced and followed for appropriate decontamination procedures between samples or sample locations, as necessary.

The following is a list of recommendations and/or requirements for effective groundwater profiling that will produce meaningful results:

- It is best if overburden stratigraphic conditions at the site can be evaluated, at least in part, prior to groundwater profiling. This will enable targeting specific zones or strata where soil grain size (and therefore groundwater flow) is most likely to be greatest, thereby increasing the chances of obtaining sufficient sample for analysis.

- The discrete sampling devices discussed herein are designed primarily for obtaining samples from granular soils, and in general, the coarser (grain size) the formation, the more readily it will yield representative groundwater samples. Fine-grained soils (fine sandy silts, silts and clays) will often clog the intake openings of the samplers, limiting the inward flow of groundwater and necessitating significant and time-consuming efforts to remove and clean the openings. This should be taken into consideration when designing a sampling program.

- It is helpful to know the depth to the water table. Obtaining a sample at that depth will maximize the ability to evaluate the potential presence of light, non-aqueous, phase liquids (LNAPL).

- The length of sample intervals should be kept to a minimum, so that analytical results are representative of a narrow vertical range. If practical sampling intervals should be less than one foot.

- Regardless of sampling interval length, it is important to accurately determine the top and bottom limits of the interval. Accurate measurements of the sampling apparatus prior to deployment is essential. In addition, during deployment of the sampling screen, the length of rod retraction must be measured.

- Avoid overlap of sampling intervals, so that analytical results apply only to that interval. Overlap of sampling intervals will cause confusion with regard to the specific location of contaminants.

- When using a sampler that requires removal after each sample, decontamination of the screen and sampling chamber is critical. In addition, the sampler must be designed with measures that prevent infiltration of borehole fluids into the sampling chamber when returning the sampler to the borehole for additional samples. In general, this requires a system of o-rings or other sealing mechanism that keeps water out until the screen is deployed at the desired depth.

- When using the Groundwater Profiler or Waterloo Sampler it is imperative that clean deionized water be circulated down through the probe and into the formation during advancement. The water must be pumped at a rate/pressure that

will prevent infiltration of groundwater into the sample screen or ports until the desired depth is reached.

These units also require sufficient purging of the deionized water prior to obtaining the groundwater sample to insure a representative formation sample is obtained. The length and diameter of the tube must be known to calculate the minimum volume of water to purge. In addition, the water that was pumped into the formation at or near the sampling depth must also be accounted for and purged out if possible.

- If the target analytes are metals, it is recommended that a PVC screen be used in lieu of stainless steel.

- With regard to sampling, be sure that the work plan or regulatory requirements do not prohibit sampling with a tubing/foot valve assembly (e.g. Waterra) due to the potential for cavitation of gases that could affect volatile organics (VOCs) analysis results. It may be necessary to use a small-diameter bailer or other means to retrieve the sample.

- Upon exploration and sampling completion, seal the borehole with grout unless it is to be completed as a permanent well installation. This will minimize or prevent the potential for the borehole to exacerbate the spread of contaminants between zones that are otherwise separated by stratigraphic barriers.

Bedrock

Water quality profiling in bedrock is generally feasible, although obtaining discrete samples in narrow zones within bedrock is generally more time consuming and costly than in overburden. However, obtaining samples in zones as narrow and frequent as in overburden is generally not practical. Bedrock groundwater profiling is generally accomplished using a system of borehole packers (or a single packer) that allows collection of a groundwater sample from an isolated interval, albeit of a generally greater length. The sample collection apparatus may or may not be similar to the overburden equipment.

The following is a partial list of considerations when performing rock corehole water quality profiling:

- The proper description and logging of bedrock core is important in determining potential target sample intervals. The presence of pits, vugs, cavities, and other

dissolution features, as well as fractures, bedding planes, and joints should be described and the correct depths of the features assigned. Rock coring should be performed in accordance with SOP 2.4, Bedrock Coring.

- Packer pressure testing, performed as a precursor to water quality profiling in the corehole, can identify potential yielding transmitting portions of the bedrock that are likely to produce sufficient water quality samples (see SOP 5.4, Packer Pressure Testing). These tests can also generate relative hydraulic conductivity values for the tested bedrock intervals. As indicated above it is crucial to recover all lost water from the formation prior to collecting representative groundwater samples.

- Development/recovery of all drilling water lost within the target interval is critical to obtaining a representative formation sample that is not diluted or contaminated by drilling water. At least as much water as was initially lost should be recovered during purging.

- It is recommended that maximum 5-foot intervals be sampled so as not to cause overlap of potential zones of interest or to isolate intervals that are too short as to include meaningful bedrock features.

- It is especially important to know the dimensions (i.e. lengths) of the apparatus being used for proper and accurate depth targeting in the bedrock.

- A submersible pump is recommended for collecting representative samples from depth. A submersible pump can operate at a lower flow rate so as to generate minimal turbulence for VOCs sampling and analysis and can collect samples from deeper intervals. Other sampling apparatus may also be used if required, based on interval depth, etc.

- To prevent cross-contamination, proper decontamination must be performed when using non-dedicated equipment that is to be used at multiple locations or at multiple depths within a single location, including packer assemblies, reusable tubing, pumps, etc.

EQUIPMENT AND VENDORS

- Geoprobe Systems
  601 N. Broadway
  Salina, Kansas 67401

(800) 436-7762

- QED Environmental Systems, Inc.
  P.O. Box 3726
  Ann Arbor, MI 48106-3726
  (800) 624-2026

- Waterloo Center for
  Groundwater Research
  University of Waterloo
  Waterloo, Ontario, Canada, N2L 3G1

- Packer assembly apparatus, if required

- Sampling pump(s) (Grundfos, peristaltic, bladder, etc.)

- Sampling apparatus (Waterra foot valve, tubing, mini-bailers, etc.)

REFERENCES

1. Company Literature, Waterloo Center for Groundwater Research, University of Waterloo, Waterloo, Ontario, Canada.

2. Company Literature, Geoprobe Systems, Salina, Kansas.

3. Company Literature, QED Environmental Systems, Inc., Ann Arbor, Michigan.

4. Company Literature, Solinst Canada, Ltd., Georgetown, Ontario, Canada.

5.8 SOIL VAPOR SAMPLING

INTRODUCTION

This procedure is for the collection of vapor samples from temporary well points for laboratory analysis in accordance with USEPA Method TO-15. This procedure includes technique for outdoor vapor collections.

PROCEDURES REFERENCED

- 1.1 Utility Clearance
- 2.1 Drilling Techniques/Background Information
- 2.2 Soil Borings
- 2.4 Soil Classification
- 2.5 Soil Vapor Point Installation
- 3.1 Well Construction Materials
- 5.8 Soil Vapor Sampling
- 7.0 Equipment Decontamination
- 8.0 Waste Characterization

SOIL VAPOR POINT INSTALLATION PROCEDURES

Operating Procedure 2.5 – Soil Vapor Monitoring Point Installation presents description of installation requirements for soil vapor point installations.

TRACER GAS TEST AND WELL PURGING

Soil Vapor well points will be sampled in order of increasing chemical concentrations (if known or anticipated). Equipment calibration, field documentation, sampling, and shipping will be conducted in accordance with the Field Sampling Plan (FSP) and Project Quality Assurance Project Plan (QAPP). Personal protective equipment will be donned in accordance with the requirements of the Project Health and Safety Plan.

1. Begin tracer gas test procedure by laying plastic sheeting at sampling location. Seal tubing and sheeting with hydrated bentonite. Fit tracer bucket over sampling location with tubing clearing bucket. Set up purge tedlar bag and GilAir pump to collect necessary purge volume.

2. Flood the bucket with commercial grade helium from a disposable compressed gas cylinder. (commercial grade can be purchased from retail stores or provided by an industrial gas supplier (i.e. welding supply).

3. Purge the installed well by connecting the well point to GilAir pump (or equivalent device) using High Density Polyethylene (HDPE) tubing and extract at least well volumes of soil vapor from the well point.

4. Using the well point installation report to obtain well depth, calculate the volume of air present in the well point, using the formula:

$$\text{Volume} = \pi\left(\frac{d}{2}\right)^2 h$$

Where,

d = the inner diameter of the tubing

h = the overall tubing length

Note: 1 in^3 = 16.39 mL

For example, ¼" O.D. tubing has an inner diameter of 0.187". Therefore a 1 foot section of that tubing has a volume of:

$$\text{Volume} = 3.14\left(\frac{0.187"}{2}\right)^2 12" * \frac{16.39\,\text{mL}}{\text{in}^3} = 5.4\,\frac{\text{mL}}{\text{foot}}$$

5. Purge the well at flow rates not to exceed 0.2 L/minute to remove three (3) volumes so that the sample collected for analysis will be representative of soil gas located in the vadose zone. GilAir pump should be calibrated to this flow rate.

6. Stop the GilAir pump and close the tedlar bag. Disconnect tedlar bag and release under He analyzer for deflection. If He is detected, modify the surface seal for the vapor sampling point and re-test. If no detection is noted, proceed with sample collection.

SOIL VAPOR COLLECTION USING SUMMA CANISTER

1. After purging, while assembling canister, close off the well point to the atmosphere using a hose clamp or similar device. The soil vapor well point is now ready to be sampled.

2. Verify the vacuum level in the passivated SUMMA® canister (or equivalent device) to be used for sample collection using the vacuum gauge supplied by the laboratory. Record the initial vacuum on the Soil Vapor Survey Sampling and Purge Information sheet (sampling record), close the valve.

3. Attach the calibrated orifice to the ¼" male fitting on the vacuum gauge using a stainless steel open end wrench. (Note: Do not over tighten. ¼ turn past finger tight should be adequate to create a proper seal.)

4. Attach tubing from soil well point to the end of the critical orifice. Be sure that the selected orifice is appropriate for the sample time length. Be sure to record any ID numbers from canister, gauge and critical orifices on chain.

5. Turn green knob on canister and allow sample collection time.

6. **MONITOR SAMPLE VACUUM DURING COLLECTION TO ENSURE THAT THE SAMPLING RATE IS CORRECT AND THAT THE CANISTER VACUUM IS NOT EXPENDED.**

7. At the conclusion of the sample collection, record the final time on the sampling record and close the valve.

8. Record final vacuum reading on chains, close the valve, remove the vacuum gauge and orifice and replace the ¼" cap on the canister inlet. Label the SUMMA® canister sample tag with proper well ID, vacuum readings and sampling time interval (e.g. 24 hours).

9. Remove the sampling train tubing apparatus for the individual well and discard. Repair soil area if possible.

10. Fill out Chain of Custody with project name, file number, sample identifications, Summa canister and related equipment serial numbers, date and time collected, and analysis requirements. Retain copies of the Chain of Custody record and relevant

shipping information. Place canisters back into their original boxes and ship them to the laboratory via an overnight courier.

- Sample number/ID
- Date and time
- Parameters to be analyzed
- Project Number
- Sampler's initials

FIELD NOTES

Field notes must document all the events, equipment used, and measurements collected during the sampling activities. *Operating Procedure 1.2* describes the data/recording procedure for field activities. The field forms should document the following for each well sampled:

- Identification of soil vapor well point location
- Soil vapor well point depth
- Static groundwater elevation and measurement technique
- Purge volume and pumping rate
- The amount of time required to purge the well point
- Purge/sampling device used
- Sample identification
- Parameters requested for analysis
- Laboratory to which samples were shipped
- Chain of custody number for shipment to laboratory
- Field observations on sampling event
- Name of sample collector(s)
- Climatic conditions including air temperature
- Problems encountered and any deviations made from the established sampling protocol.
- Sample Identification Key

EQUIPMENT AND MATERIALS REQUIRED

Soil Vapor Sampling

- SUMMA can with tag
- Critical orifice (4hr for outdoor)
- Pressure gauge
- COCs
- 40-50lbs bentonite
- Water bottle to hydrate bentonite
- Pre-made tracer test bucket

- Plastic Sheeting cut into 4 X 3 feet squares
- He canister from grocery or party store
- He detector "Ion Science Gas Check 5000is" from Ashtead rentals
- Or He detector dielectric mdg-2002 from Hazco rentals
- ¼ inch OD Tygon tubing
- Gil Air hand pump set at <.2L/min
- Tedlar bags from SKC

Extras/Misc

- Broom/dustpan for mess
- Paper towels
- Garbage bags
- Razors or pocketknife
- Nitrile gloves
- Stakes/spray paint to mark utilities COCs
- Thermometer
- Adjustable wrenches
- Tape measure
- Camera/batteries
- Watch
- FID/PID from Hazco rentals

REFERENCES

1. U.S.E.P.A., Soil Gas Sampling SOP# 2042, 1 June 1996, REV. #: 0.0

2. Compendium of Methods for the Determination of Organic Compounds in Ambient Air, EPA/625/R-96/010a, 2nd Edition, June 1999, USEPA ORD, Washington DC.

3. USEPA RCRA Groundwater Monitoring: Draft Technical guidance (EPA/530-R-93-001).

4. New York State Department of Health (NYSDOH) "Guidance for Evaluating Soil Vapor Intrusion in the State of New York", October 2006.

5. USEPA "DRAFT Guidance for Evaluating the Vapor Intrusion to Indoor Air Pathway from Groundwater and Soils (Subsurface Vapor Intrusion)", 2002.

6.0 FIELD INSTRUMENTS – USE AND CALIBRATION

6.0 FIELD INSTRUMENTS – USE AND CALIBRATION

INTRODUCTION

A significant number of field activities involve usage of electronic instruments to monitor for environmental screening and health and safety purposes. It is imperative the instruments are used and maintained properly to optimize their performance and minimize the potential for inaccuracies in the data obtained, and to insure worker's health and safety is not compromised.

This SOP provides guidance on the usage, maintenance and calibration of electronic field equipment, whether for equipment owned by the Consultant or Contractor, or equipment obtained from a rental agency.

PROCEDURES REFERENCED

- 1.2 Field Data Recording

PROCEDURE

- All monitoring equipment will be in proper working order, and operated for the purpose for which it was intended, in accordance with manufacturer's recommendations.

- Field personnel will be responsible for insuring the equipment is maintained and calibrated in the field to extent practical, or returned for office or manufacturer maintenance or calibration if warranted. Calibration is discussed in greater detail below.

- A copy of the Operating Instructions, Maintenance and Service manual and calibration log, if available, for each instrument used on a project will be kept on site at all times.

- Instruments will be operated only by personnel trained in the proper usage and calibration. In the event certification of training is required, personnel will have documentation of such certification with them on site at all times.

- Personnel must be aware that certain instruments are rated for operation within a limited range of conditions such as temperature and humidity. Usage of such instruments in conditions outside these ranges will only proceed with proper approval by a project manager and/or Health and Safety supervisor as appropriate.

- Instruments that contain radioactive source material, such as x-ray fluorescence analyzers or moisture-density gauges require specific transportation, handling and usage procedures that are generally associated with a license from the Nuclear Regulatory Commission (NRC) or an NRC-Agreement State. Under no circumstance will operation of such instruments be allowed on site unless by properly authorized and trained personnel, using the proper personal dosimetry badges or monitoring instruments.

  Calibration:

  Calibration of an electronic instrument is critical to insure it is operating properly for its intended use. Such instruments are often sensitive to changes in temperature or humidity, or chemical vapors in the working atmosphere, and as a result their response and ability to monitor conditions and provide data can change significantly.

  Calibration: Calibration of instruments shall be performed once at the beginning of every day and one additional time during the day. This includes the following parameters:

    - Frequency
    - Use of proper calibration Gases or Chemical Standards
    - Requirements for Factory Calibration

  Calibration Gas Safety: Several instruments such as photoionization detectors (PIDs), flame ionization detectors (FIDs), oxygen meters, explosimeters, combustible gas indicators and many others require use of calibration gasses contained in compressed gas cylinders. Many of these gases are combustible or explosive. Care shall be taken to minimize the potential for injury from the use of such compressed gases. Transport, handling and storage of cylinders, where necessary, shall be performed in accordance with applicable DOT regulations and site requirements.

  Calibration will only be performed in areas free of sources of spark, flame or excessive heat. Smoking will not be allowed in the vicinity of calibration gas usage areas. In situations where an extreme temperature differential exists, the unit should be brought to the temperature it is used in and calibrated at that temperature.

  Documentation of Calibration: Instrument calibration activities and maintenance activities will be documented on the appropriate field forms. In addition, protocol for documentation outlined in the Field Data Recording Procedure will be followed.

- Intrinsically Safe Requirements

Certain work locations may be such that dangerous, ignitable or explosive conditions exist. In such cases, it may be necessary to utilize only equipment that is rated as "Intrinsically Safe." Intrinsically safe instrumentation is designed with limited electrical and thermal energy levels to eliminate the potential for ignition of hazardous mixtures.

For site work requiring operation of monitoring instruments in Class I, Division I locations (as defined by the National Fire Protection Agency (NFPA)) only instrumentation rated as Intrinsically Safe will be used. Such equipment (including all accessories and ancillary equipment) must be rated to conform to Underwriter's Laboratories (UL) Standard 913, for use in a Class I, Division 1 Groups A, B, C, and D locations. It is also recommended the equipment conform with CSA Standard 22.2, No. 157-92.

- Upon completion of the field activities, equipment shall be returned to the possession of the Consultant, Contractor or Rental Agency accompanied by a written summary of any problems encountered with its use or calibration.

- Equipment shall be properly prepared for shipping, including insuring that residual gases (if applicable) are removed from the instrument, and accompanying containers of compressed gases or fluids are properly labeled and sealed.

- <u>Equipment Decontamination</u>

  Equipment that comes in contact with Site media (water level meters, water quality meters) must be cleaned **<u>before</u>** removal from the site to ensure that chemicals are not transferred to other sites. It is the responsibility of the person who requisitioned the equipment to ensure appropriate cleaning before returning the equipment. Equipment decontamination procedures are typically site-specific for unique site compounds.

EQUIPMENT

- Monitoring equipment specific to work plan tasks.
- Manufacturer's instructions, operation and maintenance information.
- Associated calibration gases, aqueous standards, etc.
- Appropriate shipping containers to facilitate transport without damage to equipment.

REFERENCE

- Underwriter's Laboratories, Inc. (http://www.ul.com/hazloc/define.htm) Standard UL 913.
- National Fire Protection Agency (http://www.nfpa.org/index.html)
- Canadian Standards Association (CSA) (http://www.csa.ca) Standard 22.2 No. 157

7.0 EQUIPMENT DECONTAMINATION

7.0 EQUIPMENT DECONTAMINATION

INTRODUCTION

This procedure describes decontamination of field equipment potentially exposed to contaminants. Proper decontamination is required to reduce the risk of transfer of contaminants from areas of contamination to other areas and to minimize the potential for cross-contamination that would compromise sample quality. The degree of decontamination required will be dependent on the nature of the activity, equipment used and on the amount of exposure to contaminants.

PROCEDURES REFERENCED

- 2.0 - Subsurface Investigations
- 4.0 - Aquifer Characterization
- 5.0 - Sample Collection for Laboratory Analysis
- 6.0 - Field Instruments – Use And Calibration
- 8.0 – Waste Characterization

PROCEDURE

A. General Procedure Discussion

Decontamination activities must be performed in a controlled area outside any exclusion zones established on the site. Care must be taken to minimize the potential for transfer of contaminated materials to the ground or onto other materials. Regardless of the size or nature of the equipment being decontaminated, the process will utilize a series of steps that involve removal of gross material (dirt, grease, oil etc.), washing with a detergent, and multiple rinsing steps. In lieu of a series of washes and rinse steps, steam cleaning with low-volume, high-pressure equipment (i.e. steam cleaner) is acceptable.

Drill rigs, backhoes and other exploration equipment, and all monitoring equipment (rented or not) in contact with the sampling media must be decontaminated prior to initiating site activities, in-between exploration locations to minimize cross-contamination potential, and prior to mobilizing off site after completion of site work. Heavy equipment is generally best decontaminated with a combination of steam-cleaning equipment and detergent scrubbing. Particular attention should be paid to parts in direct contact with contaminants, e.g. shovels, tires, augers, drilling decks, etc.

Control and containerization of all decontamination fluids is critical. A decontamination pad must be constructed that is appropriate for the size and type of equipment being

decontaminated. At a minimum, the decontamination pad will have the following elements:

- an impermeable barrier capable of containing decontaminated fluids;
- a low point where fluids will collect and can be pumped into appropriate containers;
- durability to withstand equipment such as vehicle and foot traffic;
- appropriate ancillary equipment such as racks to place decontaminated equipment to drain without further exposure to contaminated fluids;
- Labels to alert personnel as to the potential presence of contaminated materials.

B. Decontamination of Specific Sampling Equipment

The following specific decontamination procedure is recommended:

- Brush loose soil off of equipment;
- Wash equipment with laboratory grade detergent (i.e. Alconox or equivalent);
- Rinse with tap water (three rinses minimum);
- Rinse equipment with reagent grade methanol for VOC samples (this requirement may not be appropriate for sites where methanol is a contaminant of concern);
- Rinse equipment with nitric acid for metal samples (especially important for sites with potentially high metals concentrations);
- Rinse equipment with distilled water;
- Allow water to evaporate before reusing equipment; and
- Wrap equipment in aluminum foil when not being used.

C. Decontamination of Monitoring Equipment

Because monitoring equipment is difficult to decontaminate, care should be exercised to *prevent* contamination. Sensitive monitoring instruments should be protected when they are at risk of exposure to contaminants. This may include enclosing them in plastic bags allowing an opening for the sample intake. Ventilation ports should not be covered.

If contamination does occur, decontamination of the equipment will be required; however, immersion in decon fluids is not possible. As such, care must be taken to wipe the instruments down with detergent-wetted wipes or sponges, and then with deionized water-wetted wipes or sponges.

D. Disposal of Wash Solutions and Contaminated Equipment

All contaminated wash water, rinsates, solids and materials used in the decontaminated process that cannot be effectively decontaminated (such as polyethylene sheeting) will be containerized and disposed of in accordance with applicable regulations and Delphi requirements. All containers will be labeled with an indelible marker as to contents and date of placement in the container, and any appropriate stickers required (such as PCBs).

Sampling of containerized wastes will be performed immediately upon completion of the investigations to minimize storage time on site. Storage of decon wastes on site will not exceed 90 days under any circumstances.

EQUIPMENT

Decontamination equipment and solutions are generally selected based on ease of decontamination and disposability.

- Polyethylene sheeting;
- Metal racks to hold equipment;
- Soft-bristle scrub brushes or long-handle brushes for removing gross contamination and scrubbing with wash solutions;
- Large galvanized wash tubs, stock tanks, or wading pools for wash and rinse solutions;
- Plastic buckets or garden sprayers for rinse solutions;
- Large plastic garbage cans or other similar containers lined with plastic bags can be used to store contaminated clothing;
- Contaminated liquids and solids should be segregated and containerized in DOT-approved plastic or metal drums, appropriate for offsite shipping/disposal if necessary.

REFERENCE

- ASTM D5088 - Practice for Decontamination of Field Equipment Used at Non-Radioactive Waste Sites

8.0 WASTE CHARACTERIZATION – (INVESTIGATION-DERIVED WASTE)

8.0 WASTE CHARACTERIZATION (INVESTIGATION DERIVED WASTE)

RATIONALE/ASSUMPTIONS

This procedure applies when disposition of investigation soils and/or groundwater are required in accordance with the project Work Plan. Generally, this procedure is applicable to Facilities where the Project Manager has assessed the areas of investigation and has developed a waste handling plan. In some areas and/or sections within a Facility it is permitted to return soil cuttings/test pit soils and groundwater to the source area (RCRA guidance allows waste management techniques within an area of concern without "triggering" new points of waste generation). In other areas it may not be practical to return cutting/soils to their origin, and they are better handled by this characterization/disposal procedure. These practices are consistent with USEPA procedure for investigative derived wastes (IDW) at RCRA facilities and CERCLA sites.

Typically, IDW are dealt with following "Best Management Practices"; and are not handled under RCRA regulations until proven to be listed and/or identified as characteristically hazardous waste. Investigative soils and groundwater often cannot be considered a listed waste due to the lack of generator knowledge concerning chemical source, chemical origin, and timing of chemical introduction to the subsurface. Consequently, waste sampling and characterization is performed to determine if the wastes exhibit a characteristic of hazardous waste. Once the IDW characterization is complete, RCRA regulations apply if determined hazardous; if determined to be non-hazardous solid wastes, best management /solid waste handling practices apply.

9.0 SUMP INSPECTION

9.0 SUMP INSPECTION

INTRODUCTION

This procedure describes inspection of sumps. Inspection is required to reduce the risk of release of material that would compromise groundwater and soil quality. The degree of inspection will be dependent on the nature of the activity, equipment used and on the amount of material present.

BACKGROUND

1. Sumps in facilities are generally used to collect materials associated with but not limited to:
    - Machining,
    - Metalworking,
    - Coolant systems,
    - USTs
    - Wastewater
2. Sumps have no standard as to design. Engineering depends on era the sump was built. Common materials used for construction include:
    - Concrete slabs or poured in place,
    - Metal,
    - Brick,
    - Fiberglass,
    - Steel.
3. Sumps are often lined to provide additional containment of material. Common materials include:
    - Metal,
    - Plastic,
    - Coal Tar,
    - Epoxy.
4. No regulatory framework exists pertaining to general maintenance or inspection of sump integrity.

PROCEDURE

A. <u>General Procedure Discussion</u>

Inspection of the sumps must be performed in accordance with facility requirements pertaining to health and safety. Care must be taken to minimize the potential for transfer

of materials to the ground or onto other materials. Regardless of the size or nature of the sumps, the inspection will utilize a series of steps that involve removal of gross material (bulk liquid, solids, residue etc.), washing with high pressure, and inspection of the sump interior for cracking, staining, settling etc.

B. Initial Inspection Guidelines

The following specific inspection procedure is recommended:

1. Locate each sump using facility site plans and as-built drawings;
2. Inspect each sump and identify conditions and any differences from previous inspection if available. Inspection should identify:
   - Name
   - Location
   - Dimensions
   - Age
   - Estimate of volume and type of material present;
3. Remove bulk liquids, solids, sludge, and residue using dry mechanical methods consisting of hand scraping, shoveling, vacuuming or other similar means. Material removed should be containerized properly pending disposal;
4. Vacuum clean dust and particles as a final initial step;
5. Inspect empty sump for visible or noticeable cracks or settling. Photograph prior to and after cleaning.

C. Cleaning of Sump – High Pressure Washing

The high-pressure wash system utilized for the rigorous cleaning should be capable of operation from water temperatures ranging from 75° F to 180° F. The system should be capable of operation at a pressure of 3,000 to 10,000 PSI. The maximum jet reactive force should not exceed 20 pounds when operating at 10,000 PSI and at a maximum water usage rate of 4.0 GPM. At no time shall the water usage rate exceed 8.0 GPM. Splashback shall be held to a minimum and use of deflector shrouds or other means of control may be required for worker protection and/or liquid containment.

Water should be vacuumed out and the sump inspected for remaining residue, solids and presence of cracking, pitting, or settling. Photographs should be taken.

D. Disposal of Wash Solutions

All wash water, rinsates, solids and materials collected in the cleaning process (such as polyethylene sheeting) will be containerized and disposed of in accordance with applicable regulations and Delphi requirements. All containers

will be labeled with an indelible marker indicating contents and date of placement in the container, and any appropriate stickers required (such as PCBs).

Sampling of containerized wastes will be performed immediately upon completion of the investigations to minimize storage time on site. Storage of decon wastes on site will not exceed 90 days under any circumstances.

EQUIPMENT

Cleaning equipment and solutions are generally selected based on ease of use and disposability.

- Polyethylene sheeting;
- Pressure Washer;
- Long-handle brushes for removing gross product and scrubbing with wash solutions;
- Plastic buckets;
- Digital Camera;
- Large plastic garbage cans or other similar containers lined with plastic bags can be used to store contaminated clothing;
- Contaminated liquids and solids should be segregated and containerized in DOT-approved plastic or metal drums, appropriate for offsite shipping/disposal if necessary.

REFERENCE

SUMP INVENTORY FORM

Date: _____ Sump: _____
Time: _____ Location: _____
Personnel: _____ Shown on Plan: _____

Dimensions: _____ Depth: _____

Sump Construction:

- Concrete Slab ☐
- Concrete Poured ☐
- Brick ☐
- Steel ☐
- Metal ☐
- Coal Tar ☐
- Other _____

Sump Liner Material:

- N/A ☐
- Metal ☐
- Fiberglass ☐
- Plastic ☐
- Epoxy ☐
- Other ☐

Material Handled:

_____

_____

SUMP DESCRIPTION

Gravity Feed (y/n) _____
Gravity Outflow (y/n) _____ Discharged to _____
Sump Type: _____

Inverts

Description	**Dimensions**	**Location**
Example: Inflow pipe	2" diameter	180º from North, 2.3' from sump rim
A:		
B:		
C:		
D:		
E:		
F:		

APPENDIX B

EnCore Sampling Method

SW 846 METHOD 5035 FIELD SAMPLING GUIDE

En Core® Sampler Collection For Low Level Analyses (≥ 1 ug/kg)

When sampling for low level analyses, a high level sample also needs to be collected.

En Core® Sampling

Each sample point requires

- Two 5g samplers.
- One 25g sampler or one 5g sampler for screening and/or high level analysis.

(The sampler size used will be dependent on who is doing the sampling and who is doing the laboratory analysis).

- One dry weight cup.
- One T-handle.
- Paper toweling.

Procedure-Sampling

1. Remove sampler and cap from package and attach T-handle to sampler body.
2. Quickly push the sampler into a freshly exposed surface of soil until the sampler is full.
3. Use paper toweling to quickly wipe the sampler head so that the cap can be tightly attached.
4. Push cap on with a twisting motion to attach cap.
5. Fill out label and attach to sampler.
6. Repeat procedure for the other two samplers.
7. Collect dry weight sample-fill container.
8. Store samplers at 4 degrees Celsius.
9. Ship sample containers with plenty of ice to the laboratory within 40 hours of collection.

En Novative Technologies, Inc.
1795 Industrial Drive • Green Bay, WI 54302
Phone: 920-465-3960 • Toll Free: 888-411-0757 • Fax: 920-465-3963
www.ennovativetech.com

SW 846 METHOD 5035 FIELD SAMPLING GUIDE

En Core® Sampler Collection For High Level Analyses (≥ 200 ug/kg)

En Core® Sampling

Each sample point requires

- One 25g sampler or one 5g sampler (The sampler size used will be dependent on who is doing the sampling and who is doing the laboratory analysis).
- One dry weight cup.
- One T-handle.
- Paper toweling.

Procedure-Sampling

1. Remove sampler and cap from package and attach T-handle to sampler body.
2. Quickly push the sampler into a freshly exposed surface of soil until the sampler is full.
3. Use paper toweling to quickly wipe the sampler head so that the cap can be tightly attached.
4. Push cap on with a twisting motion to attach cap.
5. Fill out label and attach to sampler.
6. Collect dry weight sample-fill container.
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En Novative Technologies, Inc.
1795 Industrial Drive • Green Bay, WI 54302
Phone: 920-465-3960 • Toll Free: 888-411-0757 • Fax: 920-465-3963
www.ennovativetech.com

SW 846 METHOD 5035 FIELD SAMPLING GUIDE

Acid Preservation Sampling For Low Level Analyses (≥ 1 ug/kg)

Acid Preservation Sampling

Each sample point requires

- One 40mL VOA vial with acid preservative (for field testing of soil pH).
- Two pre-weighed 40mL VOA vials with acid preservative and stir bar (for lab analysis).
- Two pre-weighed 40mL VOA vials with water and stir bar (in case samples effervesces).
- One pre-weighed jar that contains methanol or a pre-weighed empty jar accompanied with a pre-weighed vial that contains methanol (for screening sample and/or high level analysis).
- One dry weight cup.
- One 2oz jar with NaHSO4 acid preservative (in case additional acid is needed due to high soil pH).
- One scoop capable to deliver about one gram of solid sodium bisulfate.
- pH paper.
- Weighing balance that weighs to 0.01 g (field balances may not reliably weigh to 0.01g). IAETL suggests 0.1g.
- Set of balance weights used in daily balance calibration.
- Gloves for working with pre-weighed sample vials.
- Paper toweling.

Procedure-Field Chemistry For Testing Effervescing Capacity Of Soils

1. Place ~5g of soil into a vial that contains acid preservative and no stir bar.
2. Do not cap this vial as it may EXPLODE upon interaction with the soil.
3. Observe the sample for gas evolution (due to carbonates in the soil).
4. If vigorous or sustained gas evolution occurs: then acid preservation is not acceptable to preserve the sample.
   - In this case the samples need to be collected in the VOA vials with only water and a stir bar. The vials with acid preservative CANNOT be used.
5. If a small amount or no gas evolution occurs: then acid preservation is acceptable to preserve the sample. Keep this testing vial for use in the buffering testing detailed below.
   - In this case the samples need to be collected in the VOA vials with the acid preservative and a stir bar.

Procedure-Field Chemistry For Testing Buffering Capacity Of Soils

1. If acid preservation is acceptable for sampling soils then the sample vial that was used in the effervescing testing can be used here for the buffering testing.
2. Cap the vial that contains ~5g of soil, acid preservative and no stir bar from step 1 in the effervescing testing.
3. Shake the vial gently to attempt to make a homogenous solution.
4. When done, open the vial and check the pH of the acid solution with the pH paper.
   - If the pH paper reads below 2 then the sampling can be done in the two pre-weighed 40mL VOA vials with the acid preservative and stir bar. Since the pH was below 2, it is not necessary to add additional acid to the vials.
   - If the pH paper reads above 2, then additional acid needs to be added to the sample vial.
5. Use the jar with the solid sodium bisulfate acid and add another one gram of acid to the sample.
6. Cap the vial and shake thoroughly again.
7. When done, open the vial and check the pH of the acid solution with a new piece of pH paper.
   - If the pH paper reads below 2 then the sampling can be done in the two pre-weighed 40mL VOA vials with the acid preservative and stir bar and one extra gram of acid.
   - Make a note of the extra gram of acid needed so the same amount of extra acid can be added to the vials the lab will analyze.
   - If the pH paper reads above 2, then add another gram of acid and repeat this procedure one more time.

Now that the soil chemistry has been determined the actual sampling can occur. The procedure stated below assumes the correct vials are used based on the guidance discussed.

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SW 846 METHOD 5035 FIELD SAMPLING GUIDE

Acid Preservation Sampling For Low Level Analyses (≥ 1 ug/kg)

Acid Preservation Sampling (cont.)

Procedure-Sampling

1. Wear gloves during all handling of pre-weighed vials.
2. Quickly collect a 5g sample using a cut off plastic syringe or other coring device designed to deliver 5g of soil from a freshly exposed surface of soil.
3. Carefully wipe exterior of sample collection device with clean paper toweling.
4. Quickly transfer to the appropriate VOA vial, extruding with caution so that the solution does not splash out of the vial.
5. Add more acid if necessary (this is based on the buffering testing discussed in the previous section).
6. Use the paper toweling and quickly remove any soil off of the vial threads.
7. Cap vial and weigh the jar to the nearest 0.01 g (IAETL suggests nearest 0.1g).
8. Record exact weight on sample label.
9. Repeat sampling procedure for the duplicate VOA vial.
10. Weigh the vial with methanol preservative in it to 0.01g. If the weight of the vial with methanol varies by more than 0.01 g from the original weight recorded on the vial-discard the vial. If the weight is within tolerance it can be used for soil preservation below. (IAETL suggests weighing to the nearest 0.1g)
11. Tare the empty jar or the jar that contains the methanol preservative.
12. Quickly collect a 25g or 5g sample using a cut off plastic syringe or other coring device designed to deliver 25g or 5g of soil from a freshly exposed surface of soil. The 25g or 5g size is dependent on who is doing the sampling and who is doing the laboratory analysis.
13. Carefully wipe the exterior of the collection device with clean paper toweling.
14. Quickly transfer the soil to an empty jar or a jar that contains methanol. If extruding into a jar that contains methanol be careful not to splash the methanol outside of the vial. Again, the type of jar received is dependent on who is doing the laboratory analysis.
15. If the jar used to collect the soil plug was empty before the soil was added, immediately preserve with the methanol provided using only one vial of methanol preservative per sample jar.
16. Use the paper toweling and remove any soil off of the vial threads and cap the jar.
17. Weigh the jar with the soil in it to 0.01g and record the weight on the sample label. (IAETL suggests weighing to the nearest 0.1g).
18. Collect dry weight sample-fill container.
19. Store samples at 4 degrees Celsius.
20. Ship sample containers with plenty of ice and per DOT regulations (CORROSIVE, FLAMMABLE LIQUID, POISON) to the laboratory.

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SW 846 METHOD 5035 FIELD SAMPLING GUIDE

METHANOL PRESERVATION SAMPLING FOR HIGH LEVEL ANALYSES (≥ 200 UG/KG)

METHANOL PRESERVATION SAMPLING

Each sample point requires

- One pre-weighed jar that contains methanol or a pre-weighed empty jar accompanied with a pre-weighed vial that contains methanol.
- One dry weight cup.
- Weighing balance that accurately weighs to 0.01g (field balances may not reliably weigh to 0.01g). IAETL suggests 0.1g.
- Set of balance weights used in daily balance calibration.
- Gloves for working with pre-weighed sample vials.
- Paper toweling.

Procedure-Sampling

1. Wear gloves during all handling of pre-weighed vials.
2. Weigh the vial with methanol preservative in it to 0.01g. If the weight of the vial with methanol varies by more than 0.01 g from the original weight recorded on the vial-discard the vial. If the weight is within tolerance it can be used for soil preservation/collection below (IAETL suggests weighing to the nearest 0.1g).
3. Tare the empty jar or the jar that contains the methanol preservative.
4. Quickly collect a 25g or 5g sample using a cut off plastic syringe or other coring device designed to deliver 25g or 5g of soil from a freshly exposed surface of soil. The 25g or 5g size used is dependent on who is doing the sampling and who is doing the laboratory analysis.
5. Carefully wipe the exterior of the collection device with clean paper toweling.
6. Quickly transfer the soil to an empty jar or a jar that contains methanol. If extruding into a jar that contains methanol be careful not to splash the methanol outside of the vial. Again, the type of jar used is dependent on who is doing the laboratory analysis.
7. If the jar used to collect the soil plug was empty before the soil was added, immediately preserve with the methanol provided-using only one vial of methanol preservative per sample jar.
8. Using the paper toweling-remove any soil off of the vial threads and cap the jar.
9. Weigh the jar with the soil in it to 0.01g and record the weight on the sample label. (IAETL suggest weighing to the nearest 0.1g).
10. Collect dry weight sample-fill container.
11. Store samples at 4 degrees Celsius
12. Ship sample containers with plenty of ice and per DOT regulations (CORROSIVE, FLAMMABLE LIQUID, POISON) to the laboratory.

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www.ennovativetech.com

Disposable En Core® Sampler

En Novative Technologies, Inc.
1795 Industrial Drive
Green Bay, WI 54302
Phone: 920-465-3960 • Fax: 920-465-3963
Toll Free: 888-411-0757
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Sampling Procedures

Using The En Core® T-Handle

NOTE:

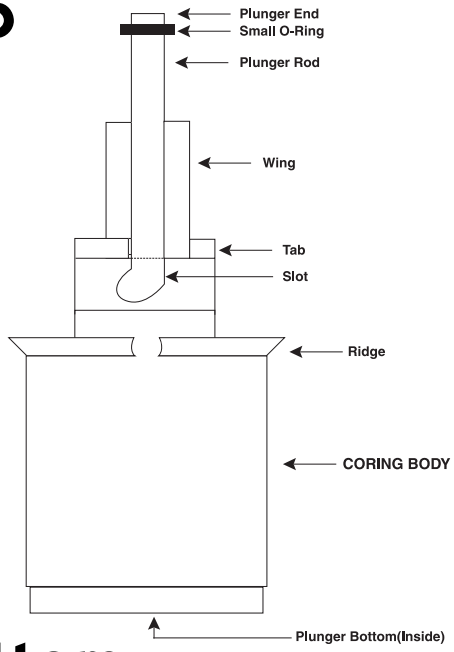
1. En Core® Sampler is a SINGLE USE device. It cannot be cleaned and/or reused.

2. En Core® Sampler is designed to store soil. Do not use En Core Sampler to store solvent or free product!

3. En Core® Sampler must be used with En Core® T-Handle and/or En Core® Extrusion Tool exclusively. (These items are sold separately.)

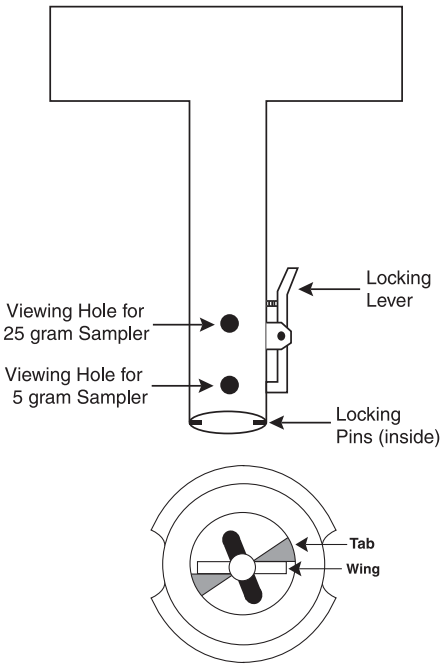
En Core®

Top



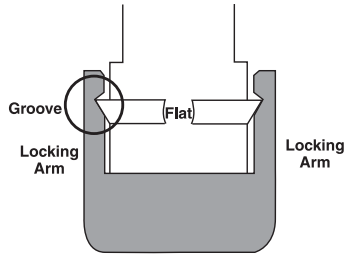
Bottom

En Core® T-Handle



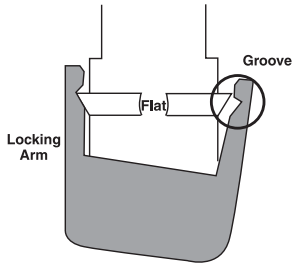
Sampler Correctly Capped

(Locking arm grooves seated over coring body ridge.)



Sampler Incorrectly Capped

(Cap appears crooked; locking arm grooves not fully seated over coring body ridge.)



BEFORE TAKING SAMPLE:

1. Hold **coring body** and push **plunger rod** down until **small o-ring** rests against **tabs**. This will assure that plunger moves freely.

2. Depress **locking lever** on En Core T-Handle. Place coring body, **plunger end first,** into open end of T-Handle, *aligning the (2)* ***slots*** *on the coring body with the (2)* ***locking pins*** *in the T-Handle*. Twist coring body clockwise to lock pins in slots. Check to ensure Sampler is locked in place. Sampler is ready for use.

TAKING SAMPLE:

3. Turn T-Handle with T-up and coring body down. This positions plunger bottom flush with bottom of coring body (ensure that plunger bottom is in position). Using T-Handle, push Sampler into soil until coring body is completely full. When full, small o-ring will be centered in T-Handle **viewing hole**. Remove Sampler from soil. Wipe excess soil from coring body exterior.

4. Cap coring body while it is still on T-handle. *Push* **cap** over **flat** area of **ridge** *and twist* to lock **cap** in place. **CAP MUST BE SEATED TO SEAL SAMPLER (see diagram)**.

PREPARING SAMPLER FOR SHIPMENT:

5. Remove the capped Sampler by depressing locking lever on T-Handle while twisting and pulling Sampler from T-Handle.

6. Lock plunger by rotating extended plunger rod fully counter-clockwise until **wings** rest firmly against tabs (see plunger diagram).

7. Attach completed tear-off label (from En Core Sampler bag) to cap on coring body.

8. Return full En Core Sampler to zipper bag. Seal bag and put on ice.

Disposable En Core® Sampler

EXTRUSION PROCEDURES

USING THE En Core® EXTRUSION TOOL

CAUTION! Always use the Extrusion Tool to extrude soil from the En Core Sampler. If the Extrusion Tool is not used, the Sampler may fragment, causing injury.

1. To attach En Core Sampler to En Core Extrusion Tool: Depress locking lever on Extrusion Tool and place Sampler, plunger end first, into open end of Extrusion Tool, aligning slots on coring body with pins in Extrusion Tool. Turn coring body clockwise until it locks into place. Release locking lever.

2. Rotate and gently push Extrusion Tool plunger knob clockwise until plunger slides over wings of coring body. (When properly positioned plunger will not rotate further.)

3. Hold Extrusion Tool with capped Sampler pointed upward so soil does not fall out when cap is removed. Remove cap from Sampler by rotating cap until locking arms are aligned with the flat area of ridge and pull cap off. To release soil core push down on plunger knob of En Core Extrusion Tool. Remove and properly dispose of En Core Sampler.

Warranty and Disclaimers

IMPORTANT: FAILURE TO USE THE EN CORE® SAMPLER IN COMPLIANCE WITH THE WRITTEN INSTRUCTIONS PROVIDED HEREIN VOIDS ALL EXPRESS AND IMPLIED WARRANTIES, INCLUDING WARRANTY OF MERCHANTABILITY AND FITNESS FOR A PARTICULAR PURPOSE.

PRINCIPLE OF USE. The En Core Sampler Cartridge System is a volumetric sampling system designed to collect, store and deliver a soil sample. The En Core Sampler comes in two sizes for sample volumes of approximately 25 or 5 grams. There are four components: the cartridge with a movable plunger; a cap with two locking arms; a T-handle (purchased separately); and an extrusion handle (purchased separately). NOTE: The En Core Sampler is designed to store soil. It is not designed to store solvent or free product.

The soil is stored in a sealed headspace-free state. The seals are achieved by three special Viton® * o-rings, two located on the plunger and one on the cap of the Sampler. At no time and under no condition should these o-rings be removed or disturbed.

QUALITY CONTROL. The cartridge is sealed in an airtight package to prevent contamination prior to use. Due to the stringent quality control requirements associated with the use of this system, the disposable cartridge is designed to be used only once.

WARRANTY. En Novative Technologies, Inc. ("En Novative Technologies") warrants that the En Core Sampler shall perform consistent with the research conducted under En Novative Technologies' approval, within thirty (30) days from the date of delivery, provided that the Customer gives En Novative Technologies prompt notice of any defect or failure to perform and satisfactory proof thereof. THIS WARRANTY DOES NOT APPLY TO THE FOLLOWING, AS SOLELY DETERMINED BY EN NOVATIVE TECHNOLOGIES: (a) Damage caused by accident, abuse, mishandling or dropping; (b)Samplers that have been opened, taken apart or mishandled; (c)Samplers not used in accordance with the directions; and (d)Damages exceeding the cost of the sampler. Seller warrants that all En Core Samplers shall be free from defects in title. THE FOREGOING WARRANTIES ARE IN LIEU OF ALL OTHER WARRANTIES, WHETHER ORAL, WRITTEN, EXPRESSED, IMPLIED OR STATUTORY, INCLUDING ANY INFORMATION PROVIDED BY SALES REPRESENTATIVES OR IN MARKETING LITERATURE. IMPLIED WARRANTIES OF FITNESS AND MERCHANTABILITY SHALL NOT APPLY. En Novative Technologies' warranty obligations and Customer's remedies, except as to title, are solely and exclusively as stated herein.

LIMITATION OF LIABILITY. IN NO EVENT SHALL EN NOVATIVE TECHNOLOGIES BE LIABLE FOR ANTICIPATED PROFITS, INCIDENTAL, SPECIAL OR CONSEQUENTIAL DAMAGES, INCLUDING, BUT NOT LIMITED TO, DAMAGES FOR LOSS OF REVENUE, DOWN TIME, REMEDIATION ACTIVITIES, REMOBILIZATION OR RESAMPLING, COST OF CAPITAL, SERVICE INTERRUPTION OR FAILURE OF SUPPLY, LIABILITY OF CUSTOMER TO A THIRD PARTY, OR FOR LABOR, OVERHEAD, TRANSPORTATION, SUBSTITUTE SUPPLY SOURCES OR ANY OTHER EXPENSE, DAMAGE OR LOSS, INCLUDING PERSONAL INJURY OR PROPERTY DAMAGE. En Novative Technologies' liability on any claim of any kind shall be replacement of the En Core Sampler or refund of the purchase price. En Novative Technologies shall not be liable for penalties of any description whatsoever. In the event the En Core Sampler will be utilized by Customer on behalf of a third party, such third party shall not occupy the position of a third-party beneficiary of the obligation or warranty provided by En Novative Technologies, and no such third party shall have the right to enforce same. All claims must be brought within one (1) year of shipment, regardless of their nature.

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The En Core™ Sampler is covered by One or More of the Following U.S. Patents: 5,343,771; 5,505,098; 5,517,868; 5,522,271. Other U.S. and Foreign Patents Pending.

* Viton® is a registered trademark of DuPont Dow Elastomers.

APPENDIX B

Quality Assurance Project Plan

QUALITY ASSURANCE PROJECT PLAN

FORMER LEEDS ASSEMBLY PLANT NORTHERN PARCEL
MOTORS LIUIDATION COMPANY
KANSAS CITY, MISSOURI

by

Haley & Aldrich, Inc.
Cleveland, Ohio

for

Motors Liquidation Company
Detroit, Michigan

December 17, 2010
File No.: 37294-001

EXECUTIVE SUMMARY

The following document outlines the scope of the quality assurance and quality control (QA/QC) activities to be performed in support of the Investigation Work Plan submitted by Motors Liquidation Company at the Former Leeds Assembly Plant Northern Parcel (Facility) in Kansas City, Missouri. This Quality Assurance Project Plan (QAPP) presents the organization, objectives, planned activities, and specific QA/QC procedures associated with the Investigation Work Plan for the Site.

Protocols for sample collection, sample handling and storage, chain-of-custody procedures, and laboratory and field analyses are described or specifically referenced to related investigation documents.

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1	Parameter List, Analytical Methods and Target Practical Quantitation Limits
2	Sample Container, Preservation Methods, Shipping, and Packaging Requirements

1. PROJECT DESCRIPTION

This Quality Assurance Project Plan (QAPP) has been prepared on behalf of Motors Liquidation Company (MLC). The QAPP is a component of the Investigation Work Plan that also includes a Field Sampling Plan (FSP), Project Management Plan (PMP), and Health and Safety Plan (HASP).

1.1 Project Objectives and Decision Statement

The primary objectives for data collection activities include:

- Define the nature and extent of potential releases of hazardous chemical constituents in environmental media (soil and groundwater) at the Facility.
- Collect sufficient data and information to evaluate the risk to human health and the environment, if any, associated with any potential releases of hazardous waste and/or hazardous constituents.

The Decision Statement for the investigation is as follows:

- Determine whether interim remedial actions are necessary to control current unacceptable risks to human health or the environment, if any; and
- Provide data for the development of a study of appropriate corrective measures (if warranted) to control current and future unacceptable risks to human health and the environment.

Associated specific objectives for field and laboratory data collection are discussed in Section 1.4 of this plan.

1.2 Site Description

The general Facility description is provided in Section 1 of the Site Investigation Work Plan and is incorporated herein by reference.

Facility Location:	Site Investigation Work Plan Figure 1 – Project Locus
Facility Plan:	Site Investigation Work Plan Figure 2 – Site Plan

1.3 Facility History

The Facility history is provided in Section 1 of the Site Investigation Work Plan and incorporated herein by reference.

Facility History:	Site Investigation Work Plan – Section 1

1.4 Project Objectives and Intended Data Use

The objective and intended data uses include:

1.4.1 Target Parameter List

The investigative program includes the sampling and analysis of environmental media for the presence of organic and inorganic constituents based on current and historical operations at the Facility. The field and laboratory parameters are summarized below and presented in Table 1.

1.4.1.1 Laboratory Parameters

The laboratory parameters include target compound list (TCL) volatile organic compounds (VOCs); TCL semi-volatile organic compounds (SVOCs); and target analyte list (TAL) metals, excluding earth metals aluminum, calcium, iron, magnesium, potassium and sodium. Table 1 of this plan present target practical quantitation limits for each compound/analyte. Method detection limit (MDL) studies have been performed by the analytical laboratory in accordance with 40 CFR Part 136, Appendix B. The results of these MDL studies are presented in Appendix D of this QAPP.

Concurrent with sample collection, several field parameters will be determined by the field sampling personnel. For soils and solid matrices, these field parameters will include visual observations, odor identification, and VOC screening using handheld monitoring equipment. For aqueous samples, at a minimum, the following parameters will be determined with field testing equipment: pH, specific conductivity, and temperature. If low-flow purging techniques are utilized for groundwater sampling, additional field parameters including turbidity, dissolved oxygen, and oxidation/reduction potential (ORP) will be measured.

1.5 Sampling Locations

The Investigation Work Plan provides a summary and rationale for the location of soil and groundwater sample. It is possible that sampling locations may change depending on the encountered field conditions. The person responsible for making such decisions will be the Field Task Coordinator and the Project Manager, whose responsibilities are described in Section 2 of this QAPP. Changes in the sampling strategy will only be implemented after approval from the MLC Project Manager.

2. PROJECT ORGANIZATION AND RESPONSIBILITIES

This section defines the roles and responsibilities of the individuals who will perform the Investigation activities. The MLC Project Manager will have the primary responsibility for implementation of the Investigatin Work Plan. The selected analytical laboratory will perform the analyses of environmental samples collected at the Site.

2.1 Management Responsibilities

A description of the project organization and responsibilities of key personnel are presented in detail in the Project Management Plan. A summary of management responsibilities follows:

Mr. David Favero – MLC Leeds Project Manager
The MLC Project Manager is responsible for implementing the project, and has the authority to commit the resources necessary to meet project objectives and requirements. The MLC Project Manager's primary function is to ensure that technical, financial and scheduling objectives are achieved successfully. The MLC Project Manager will provide the primary point of contact, control matters concerning the project, and represent the project team at regulatory agency meetings and public hearings. The MLC Project Manager will define project objectives and develop a detailed project schedule. The MLC Project Manager will establish project policy and procedures to address the specific needs of the project as a whole, as well as the objectives of each task.

Mr. David Hagen – Haley & Aldrich Project Director
The Project Director will provide final review of significant submittals to U.S. EPA and may participate in technical meetings with the U.S. EPA. The Project Director will ensure that overall technical quality is maintained. He will be actively involved in the direction of the project. The Project Director has overall responsibility for ensuring that the project meets Investigation objectives. The Project Director will perform the following:

- Acquire and apply technical and corporate resources as needed to ensure performance within budget and schedule restraints;
- Review work performed on each task to ensure quality, responsiveness and timeliness;
- Be responsible for the preparation and quality of interim and final reports; and
- Communicate with MLC about the progress of the project.

Mr. Daniel Putz – Haley & Aldrich Project Manager
The Project Manager is responsible for managing the implementation of the Investigation Work Plan and coordinating the collection of data pertaining to the Investigation. The Project Manager is responsible for technical quality control and project oversight. The Project Manager also provides approval of the QAPP.

2.2 Quality Assurance Responsibilities

The Quality Assurance team will consist of a Quality Assurance Officer (QAO) and the Project Manager. Quality Assurance responsibilities are described as follows:

Mr. Denis Conley – Haley & Aldrich Quality Assurance (QA) Officer
The QA Officer reports directly to the Project Manager and will be responsible for ensuring that QA/QC procedures are followed. The QA Officer will be responsible for overseeing the review of field and laboratory data. Additional responsibilities include the following:

- Assure the application and effectiveness of the QAPP by the analytical laboratory and the project staff; Conduct internal QA/QC of the investigation activities;
- Provide input to the Project Director and Project Manager as to corrective actions that may be required as a result of the above-mentioned evaluations;
- Prepare and review data validation and audit reports;
- Approval of the QAPP.

The QA Officer will be assisted by the data validation staff in the evaluation and validation of field and laboratory generated data.

Haley & Aldrich Data Validation Staff
The data validation staff will be independent of the laboratory and familiar with the analytical procedures performed. The validation will include a review of each validation criterion as prescribed by the guidelines presented in Section 12.2 of this document and be presented in a formal written report for submittal to the Haley & Aldrich QA Officer.

2.3 Laboratory Responsibilities

Laboratory services in support of the RFI Work Plan include the following personnel from Merit Laboratories, Inc.:

Laboratory Project Manager
The Laboratory Project Manager reports directly to the Project QA Officer and the Project Manager, and is responsible for ensuring all resources of the laboratory are available on an as-required basis. The Laboratory Project Manager also is responsible for the approval of the final analytical reports and approval of the laboratory's ability to adhere to the QAPP.

Laboratory Operations Manager
The Laboratory Operations Manager reports to the Laboratory Director. The Operations Managers work with the Project Managers and Group/Team Leaders to assure the requirements of projects are met in a timely manner. The Operations Managers will be responsible for coordinating laboratory analysis, supervising in-house chain-of-custody reports, scheduling sample analyses, overseeing data review and overseeing preparation of analytical reports.

Laboratory QA Officer
The Laboratory QA Officer works with the Project Manager and the laboratory analytical groups to review analytical data for quality and completeness. The Laboratory QA Officer also provides Case Narrative descriptions of any data quality issues encountered during the analyses conducted by the laboratory. The QA Officer also defines appropriate QA procedures, overviews QA/QC documentation and approves this QAPP.

Sample Receiving Group Leader
The Sample Receiving Group Leader reports to the Laboratory Operations Manager and works with the various groups of laboratory analysts to accomplish the following:

- Receive and inspect the incoming sample containers;
- Record the condition of the incoming sample containers;
- Sign appropriate documents;
- Verify chain-of-custody and its correctness;
- Notify the Project Manager and Operations Manager of sample receipt and inspection;
- Assign a unique identification number and customer number, and enter each into the sample receiving log;
- Initiate transfer of samples to lab sections; and
- Control and monitor access/storage of samples and extracts.

Laboratory Technical Personnel
The laboratory technical staff will have the primary responsibility in the performance of sample analysis and the execution of the QA procedures developed to determine the data quality. These activities will include the proper preparation and analysis of the project samples in accordance with the contract laboratory's Quality Assurance Manual and associated Standard Operating Procedures.

2.4 Field Responsibilities

Coordination and oversight of the field team involved in implementation of RFI Work Plan will be performed by the following personnel:

Haley & Aldrich Field QA Officer
The Field QA Officer is responsible for the overall operation of the field team and reports directly to the Project Director and MLC Project Manager. The Field QA Officer works with the project Health & Safety Coordinator to conduct operations in compliance with the project Health & Safety Plan. The Field QA Officer will facilitate communication and coordinate efforts between the Project Director and the field team members. Other responsibilities include the following:

- Develop and implement field-related work plans, ensuring schedule compliance, and adhering to management-developed project requirements;
- Coordinate and manage field staff, including sampling and drilling;
- Perform field system audits;
- Oversee quality control for technical data provided by the field staff;
- Prepare and approve text and graphics required for field team efforts;

- Coordinate and oversee technical efforts of subcontractors assisting the field team;
- Identify problems in the field; resolve difficulties in consultation with the Project Director, Project QAO, and Project Manager; implement and document corrective action procedures; and
- Participate in preparation of the final reports.

Field Team Personnel

Field Team Personnel involved in the implementation of the RFI Work Plan will be responsible for the following:

- Perform field activities as detailed in the RFI Work Plan and in compliance with the DQO's outlined in this QAPP.

- Immediately report any accidents and/or unsafe conditions to Haley & Aldrich's Site Health & Safety Coordinator and take reasonable precautions to prevent injury.

3. QUALITY ASSURANCE OBJECTIVES FOR MEASUREMENT DATA

The RFI Work Plan and associated QAPP are designed to produce data of the quality necessary to achieve the project objectives and meet or exceed the minimum standard requirements for the field and laboratory analytical methods. The overall QA objective for measurement data is to develop and implement procedures for field sampling, chain-of-custody, laboratory analysis and reporting that will provide results which are adequate for supporting the RFI statements.

The QAPP program will include the following:

- A measure of data quality in terms of precision, accuracy, representativeness, completeness, and comparability
- A mechanism for ongoing control of measurements and evaluation of data quality.

The following section is a general discussion of the criteria used to measure the field and laboratory analytical data quality.

3.1 Precision

3.1.1 Definition

Precision is defined as a quantitative measure of the degree to which two or more measurements are in agreement. Precision will be determined by collecting and analyzing field duplicate samples and by creating and analyzing laboratory duplicates from one or more of the field samples. Precision will be stated in terms of relative percent difference (RPD). The overall precision of measurement data is a mixture of sampling and analytical factors. The analytical results from the field duplicate samples will provide data on sampling precision. The results from duplicate samples created by the laboratory will provide data on analytical precision.

3.1.2 Field Precision Sample Objectives

Field precision will be assessed through collection and measurement of field duplicates at a rate of 1 duplicate per 20 investigative samples.

3.1.3 Laboratory Precision Sample Objectives

Laboratory duplicate analyses will be performed through the use of matrix spike/matrix spike duplicates (MS/MSD) for organic parameters and inorganic parameters. The analytical data will be presented in summary table format. The DQO criteria for laboratory MS/MSD analyses are provided in the associated analytical method Standard Operating Procedures (SOPs) included in Appendix C in this QAPP.

3.2 Accuracy

3.2.1 Definition

Accuracy relates to the bias in a measurement system. Bias is the difference between the observed and the "true" value. Sources of error are the sampling process, field contamination, preservation techniques, sample handling, sample matrix, sample preparation and analytical procedure limitations.

3.2.2 Field Accuracy Objectives

Sampling bias will be assessed by evaluating the results of equipment rinse and trip blanks. Equipment rinse blanks will be collected by passing ASTM Type II water over and/or through the respective sampling equipment utilized during each sampling effort.

One equipment rinse blank will be collected for each type of sampling equipment used for the sampling effort. Equipment rinse blanks will be analyzed for each target parameter for the respective sampling effort for which environmental media have been collected. (Note: If dedicated or disposable sampling equipment is used, equipment rinse samples may not be collected.)

Trip blank samples will be prepared by the laboratory and provided with each cooler that includes volatile organic compound (VOC) containers. Trip blank samples will be analyzed for each VOC for which environmental media have been collected for analysis.

3.2.3 Laboratory Accuracy Sample Objectives

Analytical bias will be assessed through the use of known laboratory control samples and site specific matrix spike sample analyses. Laboratory control samples (LCS) and MS/MSD sample analysis will be performed as prescribed by the analytical method SOPs. LCS analyses will be performed with each analytical batch of project samples to determine the accuracy of the analytical system.

One set of MS/MSD analyses will be performed with each batch of 20 project samples to assess the accuracy of identification and quantification of analytes within the site-specific sample matrices. Additional sample volume will be collected at sample locations selected for MS/MSD analyses so that method detection limits (MDL) and target practical quantitation limits can be met.

The results of the LCS and MS/MSD analyses will be presented in a summary table reporting format and evaluated versus the acceptance criteria presented in the associated analytical method SOPs. The accuracy of organic parameter analyses is also monitored through the analysis of system monitoring or surrogate compounds. Surrogate compounds are added to each sample, standard, blank, and QC samples prior to the sample preparation and analysis. Surrogate compound percent recoveries provide information on the effect of the sample matrix on the accuracy of the analyses.

3.3 Representativeness

3.3.1 Definition

Representativeness expresses the degree to which sample data accurately and precisely represent a characteristic of a population, a parameter variation at a sampling point or an environmental condition. Representativeness is a qualitative parameter that is dependent upon the design of the sampling program. The representativeness criterion is satisfied by proper selection of sampling locations and quantity of samples collected.

3.3.2 Measures to Ensure Representativeness of Field Data

Representativeness will be addressed by describing sampling techniques and the rationale used to select sampling locations. Sampling locations may be biased (based on existing data, instrument surveys, observations, etc.) or unbiased (completely random or stratified-random approaches).

For this project, sampling will generally be biased; that is, sampling associated with the soil and groundwater will be based on site knowledge or the observed presence of site contaminants. Specific sampling technique descriptions, provided as SOPs in the Field Sampling Plan, will allow consistency, repetitiveness and thus representativeness.

3.3.3 Measures to Ensure Representativeness of Laboratory Data

Representativeness in the laboratory is ensured by using proper analytical procedures and analyzing field duplicate samples. By definition, field duplicate samples are collected to be representative of a given point in space and time. Thus, sample duplicates provide both precision and representativeness information.

3.4 Completeness

3.4.1 Definition

Completeness is a measure of the amount of valid (usable) data obtained from a measuring system compared to the amount that was expected to be obtained under normal conditions. The completeness goal for all data uses is that a sufficient amount of valid data be generated so that determinations can be made related to the intended data use with a high degree of confidence.

3.4.2 Field Completeness Objectives

Completeness is a measure of the amount of valid measurements obtained from all measurements taken in this project. The field completeness objective for this project will be 90 percent or greater.

3.4.3 Laboratory Completeness Objectives

Laboratory data completeness objective is a measure of the amount of valid data obtained from all laboratory measurements. The evaluation of the data completeness will be performed at the conclusion of each sampling and analysis effort. Corrective actions, such as revised sample handling procedures, will be implemented if problems are noted.

The completeness of the data generated will be determined by comparing the amount of valid data, based on independent validation, with the total data set. The laboratory completeness goal for this project will be 90 percent or greater.

3.5 Comparability

3.5.1 Definition

Comparability is a qualitative parameter expressing the confidence with which one data set can be compared to another.

3.5.2 Measures to Ensure Comparability of Field Data

Sample data should be comparable to other measurement data for similar samples and sample conditions. This goal is achieved through using SOPs to collect, preserve, store and analyze representative samples, and the reporting of analytical results. The field SOP for the various activities to be conducted during this investigation provide guidelines to generate reproducible results and are included in Appendix B of this QAPP.

3.5.3 Measures to Ensure Comparability of Laboratory Data

Comparability of laboratory data will also be measured with the results from the analysis of Standard Reference Materials (SRM) obtained from either EPA Cooperative Research and Development Agreement (CRADA) suppliers or the National Institute of Standards and Technology (NIST) for instrument initial and continuing calibration verification. The reported analytical data will be presented in standard units of mass of contaminant within a known volume of environmental media. The units for various sample matrices are described as follows:

- Solid Matrices - milligrams (mg) contaminant per kilogram (kg) of media (dry weight).
- Aqueous Matrices – micrograms (ug) contaminant per liter (L) of media for organic analyses, and milligrams (mg) per liter (L) for inorganic analyses.
- Non-Aqueous Phase Liquids – (NAPL) – milligrams (mg) contaminant per kilogram (kg) of media.
- Gaseous Matrices – micrograms (ug) contaminant per cubic meter (M^3) of media

Additional guidance on analytical data reporting is contained in the laboratory SOPs provided in Appendix D of this QAPP.

3.6 Decision Rules

3.6.1 Definition

The decision rule is a statement that prescribes a course of action or non-action to be taken, based on assumptions to test its logical and empirical consequences.

3.6.2 Decision Rule Objective

The decision rule for the sampling and analysis data collected is as follows:

- Define statistical parameters characterizing the population (e.g., mean, maximum, and percentile) and incorporate the scale of decision making (e.g., AOI size). Samples will be collected at discrete locations and biased towards locations with the highest likelihood to encounter maximum contaminant levels to provide an indication of impacted media.

- Identify action levels (e.g., Soil Screening Levels, Risk-based Screening Levels, Preliminary Remediation Goals (PRGs)); or a reference-based standard based on a Site Conceptual Model that will be developed as part of the investigation.

- The maximum contaminant concentration or the 95^{th} percent Upper Confidence Limit of the valid data set for each parameter detected within an AOI will be used to conduct a preliminary baseline human health risks for the determination of appropriate corrective actions.

3.7 Level of Quality Control Effort

Equipment rinse, trip and method blank samples, field and laboratory duplicate samples, and laboratory control and matrix spike samples will be prepared and analyzed to determine the data quality provided by the sampling and analysis activities conducted during execution of the RFI program.

Equipment rinse blanks will be prepared by field personnel and submitted for analysis of target parameters. Equipment rinsate blanks will provide the means to assess the quality of data resulting from the field program. Equipment rinse blank samples will be analyzed to check for contamination of equipment introduced during sampling at the Site. One equipment rinse blank will be collected per sampling event per piece of non-dedicated equipment.

Trip blanks are used to assess the potential for contamination during sample storage and shipment. The trip blank consists of ASTM Type II water that has been provided with the sample containers to be used for sampling VOCs. Trip blanks will be preserved and handled in the same manner as the investigation samples. One trip blank will be included along with each shipment cooler containing project samples to be analyzed for VOCs.

Method blank samples will be prepared by the laboratory and analyzed concurrently with all project samples to assess potential contamination introduced by the laboratory during the analytical process.

Field duplicate samples are analyzed to determine sampling and analytical reproducibility. One field

duplicate will be collected for every 20 or fewer investigative samples collected for laboratory analysis or one every day.

Matrix spikes will provide information to assess the precision and accuracy of the analysis of the target parameters within the environmental media collected within an AOI. One matrix spike/matrix spike duplicate (MS/MSD) will be collected for every 20 or fewer investigative samples per sample matrix (i.e. soil or groundwater). Soil MS/MSD samples require 3X sample volume for VOCs only. Aqueous MS/MSD samples require 3X sample volume for VOC analysis and 2X sample volume for the remaining parameters.

4. SAMPLING PROCEDURES

Samples of groundwater and soil will be obtained during implementation of the Investigation Work Plan. The sampling procedures for the Investigation Work Plan will be consistent for the objectives of this QAPP. The Field Sampling Plan (FSP) describes each of the sampling tasks and objectives.

Refer to the FSP for the field investigation SOPs for sampling of environmental media.

4.1 Sample Containers

Sample containers for each sampling task will be provided by the project laboratory. The containers will be cleaned by the manufacturer to meet or exceed the analyte specifications established in the U.S. EPA, "Specifications and Guidance for Obtaining Contaminant-Free Sample Containers," April 1992, OSWER Directive #9240.0-0.5A. Certificates of analysis for each lot of sample containers used during the Investigation will be maintained by the laboratory and will be available upon request.

The appropriate sample containers, preservation method, maximum holding times, and shipping information for each target parameter and sampling task are provided in Table 2 of this QAPP.

4.2 Sample Labeling

Each sample will be labeled with a unique sample number that will facilitate tracking and cross-referencing of sample information. Equipment rinse blank and field duplicate samples also will be numbered with a unique sample number to prevent analytical bias of field QC samples.

Refer to the FSP for the sample labeling procedures.

4.3 Field QC Sample Collection

4.3.1 Equipment Rinse Blank Sample Collection

Equipment rinse blank samples will be collected when non-dedicated sampling equipment is used to collect samples. Equipment rinse blanks consist of distilled water that has been routed through decontaminated sampling equipment and collected into the appropriate containers. The containers will be filled in order of decreasing analyte volatility (i.e., VOCs first, SVOCs second, followed by the containers for the remaining analyses).

4.3.2 Field Duplicate Sample Collection

4.3.2.1 Water Samples

Field duplicate samples will be collected using the following procedure:

1. The first sample container is filled to the proper level and sealed. The procedure is repeated for the second sample container.

2. The samples are properly labeled as specified in the Section 4.2 of this QAPP and the FSP.

3. Steps 1 and 2 are repeated for the bottles for each analysis. The samples are collected in order of decreasing analyte volatility as detailed in Section 4.3.1.

4. Chain-of-custody documents are executed.

5. The samples are packaged as specified in Table 2 of this QAPP.

4.3.2.2 Soil Samples

Soil field duplicates will be collected as specified in the following:

1. The split-spoon sampler or trowel will be retrieved from the sampling point.

2. Soil for VOC analysis will be removed from the sampling device utilizing a specialized zero headspace sampling tool (i.e. EnCore Sampler™) as specified in the FSP.

3. Soil for non-VOC analysis will be removed from the sampling device and placed in a stainless steel mixing bowl. The soil will be thoroughly homogenized using stainless steel utensils and the sample containers will be filled in order of decreasing analyte volatility as described in Section 4.3.1.

5. CUSTODY PROCEDURES

Custody is one of several factors necessary for the admissibility of environmental data as evidence in a court of law. Custody procedures help to satisfy the two major requirements for admissibility: relevance and authenticity. Sample custody is addressed in three parts: field sample collection, laboratory analysis and final project files. Final project files, including all originals of laboratory reports, are maintained under document control in a secure area.

Custody of a sample begins when the sample is collected by or transferred to an individual and ends when that individual relinquishes or disposes of the sample. A sample or project file is under custody if:

- the item is in actual possession of a person;
- the item is in the view of the person after being in actual possession of the person;
- the item was in actual possession and subsequently stored to prevent tampering; or
- the item is in a designated and identified secure area.

5.1 Field Custody Procedures

Field personnel will keep written records of field activities on applicable preprinted field forms or in a bound field notebook. The written records provide the means of recording data collecting activities. These records will be written legibly in ink and will contain pertinent field data and observations. Entry errors or changes will be crossed out with a single line, dated and initialed by the person making the correction.

The beginning of each entry in the logbook or preprinted field form will contain the following information:

- Date,
- Start time,
- Weather,
- Names of field personnel (including subcontractors),
- Level of personal protective equipment used at the Site, and
- Names of all visitors and the purpose of their visit.

For each measurement and sample collected, the following will be recorded:

- Detailed description of sample location,
- Equipment used to collect sample or make measurement and the date equipment was calibrated,
- Time sample was collected,
- Description of the sample conditions,
- Depth sample was collected (if applicable),
- Volume and number of containers filled with the sample; and,
- Sampler’s name.

5.1.1 Field Procedures

Data quality can be affected by sample collection activities. If the integrity of collected samples is questionable, the data, regardless of its analytical quality, will also be questionable.

The following procedure describes the process to maintain the integrity of the samples:

- Upon collection, samples are placed in the proper containers. In general, samples collected for organic analysis will be placed in pre-cleaned glass containers and samples collected for inorganic analysis will be placed in pre-cleaned plastic (polyethylene) bottles. Refer to the FSP for sample packaging procedures.

- Samples will be assigned a unique sample number and a sample label will be affixed. Refer to the FSP for sample labeling procedures.

- Samples will be properly and appropriately preserved by field personnel in order to minimize loss of the constituent(s) of interest due to physical, chemical or biological mechanisms. Sample aliquots that require preservation (pH adjustment) will be checked with pH paper at the time of preservation. If required, additional preservative will be added until proper preservation is achieved. Aqueous samples for VOC analysis will be collected in pre-preserved containers and will not undergo this procedure.

- Appropriate volumes will be collected to ensure that the appropriate estimated quantitation limits can be achieved and the required QC sample analyses can be performed.

5.1.2 Transfer of Custody and Shipment Procedures

- A chain-of-custody (COC) record will be completed at the time of sample collection and will accompany each shipment of project samples to the laboratory. The field personnel collecting the samples will be responsible for the custody of the samples until the samples are relinquished to the laboratory. Sample transfer will require the individuals relinquishing and receiving the samples to sign, date and note the time of sample transfer on the COC record.

- Samples will be shipped or delivered in a timely fashion to the laboratory so that holding-times and/or analysis times as prescribed by the methodology can be met.

- Samples will be transported in containers (coolers) which will maintain the refrigeration temperature for those parameters for which refrigeration is required in the prescribed preservation protocols. Water VOC samples will be placed in bubble wrap bags while soil VOC samples will be placed in sealed sample pouches. The remaining samples in glass containers will be wrapped in bubble wrap and placed in the sample cooler. Samples in polyethylene containers will be placed upright directly in the sample cooler.

- Samples will be placed in an upright position and limited to one layer of samples per cooler. Additional bubble wrap or packaging material will be added to fill the cooler.

Shipping containers will be secured with strapping tape and custody tape for shipment to the laboratory. Refer to the FSP for custody and shipment procedures.

- When samples are split with the U.S. EPA representatives, a separate chain-of-custody will be prepared and marked to indicate with whom the samples are shared. The person relinquishing the samples to the U.S. EPA will require the representative's signature acknowledging sample receipt.

- If samples are sent by a commercial carrier, a bill of lading will be used. A copy of the bill of lading will be retained as part of the permanent record. Commercial carriers will not sign the custody record as long as the custody record is sealed inside the sample cooler and the custody tape remains intact.

- Samples will be picked up by a laboratory courier or transported overnight by a courier to the laboratory the same day they are collected unless collected on a weekend or holiday. In these cases, the samples will be stored in a secure location until delivery to the lab. Additional ice will be added to the cooler as needed to maintain proper preservation temperatures.

5.2 Laboratory Chain-of-Custody Procedures

A sample custodian will be assigned the responsibility of sample control. It will be the responsibility of the sample custodian to receive all incoming samples. Once received, the custodian will document that each sample has been received in good condition (i.e., unbroken, cooled, etc.) and that the associated paperwork, such as chain-of-custody forms, have been completed. The custodian will sign the chain-of-custody forms. In special cases, the custodian will document from appropriate sub-samples that chain-of-custody with proper preservation has been accomplished. The custodian will also document that sufficient sample volume has been received to complete the analytical program. The sample custodian will then place the samples into secure, limited access storage (refrigerated storage, if required). The sample custodian will assign a unique number to each incoming sample for use in the laboratory. The unique number will then be entered into the sample-receiving log. The laboratory date of receipt will also be noted.

Consistent with the analyses requested on the chain-of-custody form, analyses by the laboratory's analysts will begin in accordance with the appropriate methodologies. Samples will be removed from secure storage only after internal chain-of-custody sign-out procedures have been followed.

5.3 Storage of Samples

Unused portions of sample bottles will be returned to secure storage after analysis. Upon completion of the analytical work, samples will be disposed of by the sample custodian. The length of time that samples are held will be at least thirty (30) days after reports have been submitted. Disposal of remaining samples will be completed in compliance with Federal, State and local requirements.

5.4 Final Project Files Custody Procedures

The final project files will be the central repository for all documents pertaining to sampling and analysis activities as described in this QAPP. The Haley & Aldrich Project Manager will be the custodian of the project file. The project files for the Investigation, including all relevant records, reports, logs, field notebooks, pictures, subcontractor reports and data reviews will be maintained in a secure area under custody of the Project Director or his designee.

The final project file will include the following:

- Project plans and drawings,
- Field data records,
- Sample identification documents and soil boring/monitoring well logs,
- Chain-of-custody documentation,
- Correspondence,
- References, literature,
- Laboratory data deliverables,
- Data validation and assessment reports
- Progress reports, QA reports, and
- Final report.

The laboratory will be responsible for maintaining analytical logbooks, laboratory data and sample chain of custody documents. Raw laboratory data files and copies of hard copy reports will be inventoried and maintained by the laboratory for a period of five years at which time the laboratory will contact the Haley & Aldrich Project Manager regarding the disposition of the project files.

6. CALIBRATION PROCEDURES AND FREQUENCY

6.1 Field Instrument Calibration Procedures

Several field instruments will be used for both on-site screening of samples and for health and safety monitoring, as described in the Health and Safety Plan (HASP). On-site monitoring for health and safety purposes may be accomplished using several different organic vapor detection devices, such as an Organic Vapor Analyzer (OVA), Combustible Gas Indicator (CGI), and/or Draeger tubes.

Field instruments will be calibrated at the beginning of each day and checked at least once during field activities to verify performance. Instrument specific calibration procedures will be performed in accordance with the instrument manufacturer's requirements as provided in the FSP.

6.2 Laboratory Instrument Calibration Procedures

Reference materials of known purity and quality will be utilized for the analysis of environmental samples. The laboratory will carefully monitor the preparation and use of reference materials including solutions, standards and reagents through well-documented procedures.

All solid chemicals and acids/bases used by the laboratory will be rated as "reagent grade" or better. All gases will be "high" purity or better. All Standard Reference Materials (SRMs) or Performance Evaluation (PE) materials will be obtained from approved vendors of the National Institute of Standards and Technology (formerly National Bureau of Standards), the U.S. EPA Environmental Monitoring Support Laboratories (EMSL), or reliable Cooperative Research and Development Agreement (CRADA) certified commercial sources.

Parameter specific calibration procedures are provided in the laboratory SOPs presented in Appendix C of this QAPP.

7. ANALYTICAL PROCEDURES

Analytical procedures to be utilized for analysis of environmental samples will be based on referenced U.S. EPA analytical protocols and/or project specific SOPs provided as an attachment to the FSP.

7.1 Field Analytical Procedures

Field analytical procedures include the measurement of pH, temperature, and specific conductivity during sampling of groundwater, and the qualitative measurement of volatile organic compounds (VOCs) during the collection of soil samples. Specific guidance in the measurement of these parameters is presented in the SOPs provided in Appendices C of this QAPP.

7.2 Laboratory Analytical Procedures

Laboratory analyses will include the measurement of Target Compounds List (TCL) VOCs, TCL Semi-volatile Organic Compounds (SVOCs), TCL Polychlorinated Biphenyl (PCBs), and Target Analyte List (TAL) metals based on the U.S. EPA methodology requirements promulgated in:

- "Test Methods for Evaluating Solid Waste," SW-846 EPA, Office of Solid Waste, and promulgated updates, 1986.

The laboratory reporting limits (RL) and associated method detection limits (MDLs) for the target analytes and compounds for the environmental media to be analyzed as part of the investigation are presented in Table 1 of this QAPP.

7.2.1 List of Project Target Compounds and Laboratory Reporting Limits

A complete list of project target compounds and reporting limits for each analyte is listed in Table 1. MDL have been experimentally determined by the project laboratory using the method provided in 40 CFR, Part 136 Appendix B.

7.2.2. List of Method Specific Quality Control (QC) Criteria

The laboratory SOPs included in this QAPP contain a section that presents the minimum QC requirements for the project analyses. QAPP Section 8 references the frequency of the associated QC samples for each sampling effort and matrix.

8. INTERNAL QUALITY CONTROL CHECKS

This section presents the internal quality control checks that will be employed for field and laboratory measurements.

8.1 Field Quality Control

8.1.1 Equipment Rinse Blanks

Internal quality control checks will include analysis of equipment blanks to validate equipment cleaning activities. Whenever possible, dedicated equipment will be employed to reduce the possibility of cross-contamination of samples.

An equipment rinse sample will be prepared for each type of sampling equipment on which decontamination procedures have been performed as part of each sampling event.

8.1.2 Trip Blanks

Trip blanks samples will be prepared by the project laboratory using ASTM Type II or equivalent water placed within pre-cleaned 40 milliliter (ml) VOC vials equipped with teflon septa. Trip blanks will accompany each sample delivery group (SDG) of environmental samples collected for analysis of VOCs.

Trip blank samples will be placed in each cooler used to store and transport project samples to be analyzed for VOCs.

8.2 Laboratory Procedures

Procedures which contribute to maintenance of overall laboratory quality assurance and control include appropriately cleaned sample containers, proper sample identification and logging, applicable sample preservation, storage and analysis within prescribed holding times, and use of controlled materials.

8.2.1 Field Duplicate Samples

The precision or reproducibility of the data generated will be monitored through the use of field duplicate samples. Field duplicate analysis will be performed at a frequency of 1 in 20 project samples.

Precision will be measured in terms of the absolute value of the relative percent difference (RPD) as expressed by the following equation:

$$RPD = \frac{\left| \frac{R1 - R2}{(R1 - R2)} \right|}{2} X100\%$$

Acceptance criteria for duplicate analyses performed on solid matrices will be 50% and aqueous matrices will be 30%. RPD values outside these limits will require an evaluation of the sampling and/or analysis procedures by the project QA Officer and/or laboratory QA Director. Corrective actions may include re-analysis of additional sample aliquots and/or qualification of the data.

8.2.2 Matrix Spike Samples

Five percent (1 sample set per 20 project samples) of each project sample matrix for each analytical method performed will be spiked with known concentrations of the specific target compounds/analytes.

The amount of the compound recovered from the sample compared to the amount added will be expressed as a percent recovery. The percent recovery of an analyte is an indication of the accuracy of an analysis within the site-specific sample matrix. Percent recovery will be calculated for MS/MSD using the following equation.

$$\%\ Recovery = \frac{Spiked\ Sample - Background}{Known\ Value\ of\ Spike}\ x\ 100\%$$

Current laboratory Quality Control limits for matrix spike (MS) recoveries are provided in Appendix C.

If the quality control value falls outside the control limits (UCL or LCL) due to sample matrix effects, the results will be reported with appropriate data qualifiers. To determine the effect non-compliant MS recoveries have on the reported results, the recovery data will be evaluated as part of the validation process.

8.2.3 Laboratory Control Sample (LCS) Analyses

The laboratory will perform LCS analyses prepared from Standard Reference Materials (SRM). The SRM will be supplied from an independent manufacturer and traceable to NIST materials with known concentrations of each target analyte to be determined by the analytical methods performed. In cases where an independently supplied SRM is not available, the LCS may be prepared by the laboratory from a reagent lot other than that used for instrument calibration.The laboratory will evaluate LCS analyses in terms of percent recovery using laboratory generated control limits.

LCS recoveries that do not meet acceptance criteria will be deemed invalid. Analysis of project samples will cease until an acceptable LCS analysis has been performed. If sample analysis is performed in association with an out-of-control LCS sample analysis, the data will be deemed invalid.

Corrective actions will be initiated by the Haley & Aldrich QA Officer and/or laboratory QA officer to investigate the problem. After the problem has been identified and corrected, the solution will be noted in the instrument run logbook and re-analysis of project samples will be performed, if possible.

The analytical anomaly will be noted in the sample delivery group (SDG) Case Narrative and reviewed by the data validator. The data validator will confirm that appropriate corrective actions were implemented and recommend the applicable use of the affected data.

8.2.4 Surrogate Compound/Internal Standard Recoveries

For VOCs, surrogates will be added to each sample prior to analysis to establish purge and trap efficiency. Quantitation will be accomplished via internal standardization techniques.

The recovery of surrogate compounds and internal standards will be monitored by laboratory personnel to assess possible site-specific matrix effects on instrument performance.

For semi-volatile organics, surrogates will be added to the raw sample to assess extraction efficiency. Internal standards will be added to all sample extracts and instrument calibration standard immediately before analysis for quantitation via internal standardization techniques.

Method specific quality control (QC) limits are provided in the laboratory method SOPs in Appendix C of this QAPP. Surrogate compound/internal standard recoveries that do not fall within accepted QC limits for the analytical methodology performed will have the analytical results flagged with data qualifiers as appropriate by the laboratory and will be noted in the laboratory report Case Narrative.

To ascertain the effect non-compliant surrogate compound/internal standard recoveries may have on the reported results, the recovery data will be evaluated as part of the validation process. The data validator will provide recommendations for corrective actions including but not limited to additional data qualification.

8.2.5 Calibration Verification Standards

As presented in Section 7.0 of this QAPP, calibration verification (CV) standards will be utilized to confirm instrument calibrations and performance throughout the analytical process. CV standards will be prepared as prescribed by the respective analytical protocols. Continuing calibration will be verified by compliance with method-specific criteria prior to additional analysis of project samples. Non-compliant analysis of CV standards will require immediate corrective action by the project laboratory QA officer and/or designated personnel.

Corrective action may include re-analysis of each affected project sample, a detailed description of the problem, the corrective action undertaken, the person who performed the action, and the resolution of the problem.

8.2.6 Laboratory Method Blank Analyses

Method blank sample analysis will be performed as part of each analytical batch for each methodology performed. If target compounds are detected in the method blank samples, the reported results will be flagged by the laboratory in accordance with standard operating procedures. The data validator will provide recommendations for corrective actions including but not limited to additional data qualification.

9. DATA REDUCTION, VALIDATION AND REPORTING

All data generated through field activities or by the laboratory operation shall be reduced and validated prior to reporting in accordance with the following procedures:

9.1 Data Reduction

9.1.1 Field Data Reduction Procedures

Field data reduction procedures will be minimal in scope compared to those implemented in the laboratory setting. The pH, conductivity, temperature, turbidity and VOC readings collected in the field will be generated from direct read instruments. These data will be written into field logbooks immediately after measurements are taken. If errors are made, data will be legibly crossed out, initialed and dated by the field member, and corrected in a space adjacent to the original entry.

9.1.2 Laboratory Data Reduction Procedures

Laboratory data reduction procedures will be implemented as described in the appropriate chapter of SW-846, Third Edition. All calculations will be checked at the conclusion of each day. Errors will be noted; corrections will be made with the original notations crossed out legibly. Analytical results for soil samples shall be calculated and reported on a dry weight basis.

Quality control data (e.g., laboratory duplicates, surrogates, matrix spikes, and matrix spike duplicates) will be compared to the method acceptance criteria. Data considered to be acceptable will be entered into the laboratory information management system (LIMS). Data summaries will be sent to the Laboratory QA Officer for review. Unacceptable data shall be appropriately qualified in the project report. Case narratives will be prepared which will include information concerning data that fell outside acceptance limits and any other anomalous conditions encountered during sample analysis.

9.2 Data Validation

Data validation procedures shall be performed for both field and laboratory operations as described below:

9.2.1 Procedures Used to Evaluate Field Data

Procedures to evaluate field data for this project will include review of field logbooks and checking for transcription errors to project specific documents. This task will be the responsibility of the QAO and the Project Manager.

9.2.2 Procedures to Validate Laboratory Data

Validation of the analytical data will be performed by the Haley & Aldrich QAO or designee using the following documents as guidance for the review process:

"U.S.EPA Contract Laboratory Program National Functional Guidelines for Organic Data Review," EPA-540-R-08-01, June 2008 and the "U.S. EPA Contract Laboratory Program National Functional Guidelines for Inorganic Data Review," EPA-540-R-04-004, October 2004.

The specific data qualifiers used will be as presented and defined in the U.S. EPA National Functional Guidelines. Validation will be performed by qualified personnel at the direction of the Haley & Aldrich QAO. Data review and validation will consist of two tiers of assessment.

Tier I data validation will be performed on 100% of the laboratory quality control summary data deliverables. The following will be evaluated:

Organic Analyses

- technical holding times;
- GC/MS instrument performance check;
- initial and continuing calibration;
- internal standard performance;
- method, trip and equipment rinsate blanks;
- system monitoring compounds (surrogate spikes);
- MS/MSD results;
- laboratory control samples; and
- field duplicate samples.

Inorganic Analyses

- technical holding times;
- blanks;
- laboratory control samples;
- MS/MSD results; and
- field duplicate.

Tier II validation will be completed on 10 % of the expanded deliverables during the initial investigation activities. The following will be evaluated during tier II validation:

Organic Analyses

- technical holding times;
- GC/MS instrument performance check;
- initial and continuing calibration;
- blanks;
- system monitoring compounds (surrogate spikes);
- MS/MSD results;
- laboratory control samples;
- internal standard performance;
- system performance;
- retention time windows (GC analyses); and

- field duplicates

Inorganic Analyses

- technical holding times;
- initial and continuing calibration;
- blanks;
- interference check samples;
- laboratory control samples;
- matrix duplicate sample analysis;
- matrix spike sample analysis;
- ICP interference check sample;
- ICP serial dilution;
- ICP/MS internal standard performance;
- sample result verification; and
- field duplicates.

The completeness of each data package will be evaluated by the data validation staff. Completeness checks will be administered on all data to determine whether deliverables specified in the QAPP are present. At a minimum, deliverables will include sample chain-of-custody forms, analytical results, QC summaries and supporting raw data from instrument printouts. The reviewer will determine whether all required items are present and request copies of missing deliverables.

9.3 Data Reporting

Data reporting procedures shall be carried out for field and laboratory operations as indicated below:

9.3.1 Field Data Reporting

Field data reporting shall be conducted principally through the transmission of report sheets containing tabulated results of all measurements made in the field and documentation of all field calibration activities.

9.3.2 Laboratory Data Reporting

The laboratory data reporting package will be sufficient to perform a data validation in accordance with protocols described above. The task of reporting laboratory data will be initiated after the completion of data validation.

In addition to the record of the chain-of-custody, the final laboratory data report format shall consist of the following:

QC Data Summary Package

- Case Narrative;

- laboratory analysis performed;
- any deviations from intended analytical strategy;
- laboratory batch number;
- number of samples and respective matrices;
- QC procedures utilized and also references to the acceptance criteria;
- laboratory report contents;
- project name and number;
- condition of samples "as received";
- discussion of whether or not sample holding times were met;
- discussion of technical problems or other observations which may have created analytical difficulties;
- discussion of laboratory QC checks which failed to meet project criteria;
- signature of the Laboratory QA Officer or his designee; and
- CLP Data Summary Forms I-IX (or equivalent).

Sample Data Package

- Case narrative for each analyzed batch of samples;
- cross referencing of laboratory sample to project sample identification numbers;
- definition of data qualifiers to be used;
- methods of sample preparation and analyses for samples;
- sample results;
- raw data for sample results and laboratory quality control samples;
- results of initial and continuing calibration checks and GC/MS tuning results;
- matrix spike and matrix spike duplicate recoveries, laboratory control samples, method blank results, surrogate recoveries, calibration check compounds and system performance check compound results (organics);
- matrix spike recoveries and matrix duplicate relative percent differences, laboratory control sample, serial dilutions, method blank results, and reagent blank results (inorganics); and

- labeled and dated chromatograms/spectra of sample results and laboratory quality control checks.

9.3.3 Validation Reporting

Validation findings will be reported in a Data Useability Summary Report (DUSR). The DUSR will summarize the findings of items/criteria applicable to the analysis of project samples and associated QA/QC procedures under the tiered assessment approach as described in Section 9.2.2. One DUSR will be produced for each Data Package produced by the laboratory.

The validator will coordinate with the project assigned database program manager so that the Site database is updated with validator qualified data. Only validator qualified data will be used for analysis or presented on final tables and figures.

10. PERFORMANCE AND SYSTEM AUDITS

A performance audit is an independent quantitative comparison with data routinely obtained in the field or the laboratory. Performance audits include two separate, independent parts: internal and external audits.

10.1 Field Performance and System Audits

10.1.1 Internal Field Audit Responsibilities

Internal audits of field activities include the review of sampling and field measurements conducted by the QA Officer. Internal field audits will be conducted once during each phase of the sampling and at the conclusion of the project.

The audits will include examination of the following:

- Field sampling records, screening results, instrument operating records,
- Sample collection,
- Handling and packaging in compliance with procedures,
- Maintenance of QA procedures, and
- Chain-of-custody reports.

Follow up audits will be conducted to correct deficiencies and to verify that procedures are maintained throughout the investigation.

An example field audit checklist is included in Appendix E of this QAPP.

10.1.2 External Field Audit Responsibilities

External audits may be conducted by the Project Manager at any time during the field operations. These audits may or may not be announced and are at the discretion of the U.S. EPA Project Staff. The external field audits can include (but are not limited to) the following:

- Sampling equipment decontamination procedures,
- Sample bottle preparation procedures,
- Sampling procedures,
- Examination of health and safety plans,
- Procedures for verification of field duplicates, and
- Field screening practices.

10.2 Laboratory Performance and System Audits

10.2.1 Internal Laboratory Audit Responsibilities

The laboratory system audits will be conducted by the Laboratory QA Officer or designee and performed on an annual basis. Laboratory performance audits will be conducted prior to field activities or on an annual basis, depending on the duration of the project.

The system audit will include an examination of laboratory documentation including sample receiving logs, sample storage, chain-of-custody procedures, sample preparation and analysis, and instrument operating records. System audits, as opposed to performance audits, are strictly qualitative and consist of an on-site review of a laboratory's quality assurance system and physical facilities for calibration and measurement.

At the conclusion of internal or external system audits, reports will be provided to the laboratory's operating divisions for appropriate comment and remedial/corrective action where necessary. Written response to internal as well as external audits will be required. Records of audits and corrective actions will be maintained by the Laboratory QA Officer.

10.2.2 External Laboratory Audit Responsibilities

External audits will be conducted as required, by appropriate QA personnel of the U.S. EPA. External audits may include any of the following:

- Review of laboratory analytical procedures,
- Laboratory on-site visits, and
- Submission of performance evaluation samples for analysis.

Failure of any of the above audit procedures can lead to laboratory disqualification, and another suitable laboratory will have to be chosen. An on-site review can consist of:

- Sample receipt procedures,
- Custody, sample security and log-in procedures
- Review of instrument calibration logs,
- Review of QA procedures,
- Review of log books,
- Review of analytical SOPs, and
- Personnel interviews.

Review of a data package from samples recently analyzed by the laboratory can include (but not be limited to) the following:

- Comparison of resulting data to the SOP or method,
- Verification of initial and continuing calibrations within control limits,
- Verification of surrogate recoveries and instrument timing results,
- Review of extended quantitation reports for comparisons of library spectra to instrument spectra, where applicable, and
- Assurance that samples are run within holding times.

11. PREVENTIVE MAINTENANCE

11.1 Field Instrument Preventive Maintenance

The field equipment preventive maintenance program is designed to ensure the effective completion of the sampling effort and to minimize equipment down time. Program implementation is concentrated in three areas:

- Maintenance responsibilities,
- Maintenance schedules, and
- Inventory of critical spare parts and equipment.

The maintenance responsibilities for field equipment will be assigned to the task leaders in charge of specific field operations. Field personnel will be responsible for daily field checks and calibrations and for reporting any problems with the equipment. The maintenance schedule will follow the manufacturer's recommendations. In addition, the field personnel will be responsible for determining that critical spare parts are included with the field equipment. An adequate inventory of spare parts will be maintained. The inventory will primarily contain parts that are subject to frequent failure, have limited useful lifetimes and/or cannot be obtained in a timely manner.

11.2 Laboratory Instrument Preventive Maintenance

Analytical instruments at the laboratory will undergo routine and/or preventive maintenance. The extent of the preventive maintenance will be a function of the complexity of the equipment.

Generally, annual preventive maintenance service will involve cleaning, adjusting, inspecting and testing procedures designed to deduce instrument failure and/or extend useful instrument life. Between services, routine operator maintenance and cleaning will be performed according to manufacturer's specifications by laboratory personnel.

Maintenance records will be placed on file at the laboratory and will be made available upon request.

12. SPECIFIC ROUTINE PROCEDURES USED TO ASSESS DATA PRECISION, ACCURACY, AND COMPLETENESS

12.1 Field Measurements

Field-generated information will be reviewed for validity. The review will be performed by the Field QA Officer and will typically include evaluation of bound logbooks/forms, data entry and calculation checks. Field data will be assessed by the Project Field QA Officer who will review the field results for compliance with the established QC criteria that are specified in Section 3 of this QAPP. The accuracy of pH and specific conductance will be assessed using daily instrument calibration, calibration check, and blank data. Accuracy and precision of the soil VOC screening will be determined using duplicate readings of calibration checks. Field data completeness will be calculated using the following equation:

$$\% \text{ Completeness} = \frac{\text{Valid (usable) Data Obtained}}{\text{Total Data Planned}} \text{ X } 100$$

12.2 Laboratory Data

Surrogate, internal standard and matrix spike recoveries will be used to evaluate data quality. The laboratory quality assurance/quality control program will include the following elements:

- Precision, in terms of relative percent difference (RPD), will be determined by relative sample analysis at a frequency of one duplicate analysis for each batch of 20 project samples or a frequency of 5%. RPD is defined as the absolute difference of duplicate measurements divided by the mean of these analyses normalized to percentage.

- Accuracy, in terms of percent recovery (recovery of known constituent additions or surrogate recoveries), will be determined by the analysis of spiked and unspiked samples. MS/MSD will be used to determine analytical accuracy. The frequency of MS/MSD analyses will be one project sample MS/MSD per set of twenty project samples.

- One method blank will be prepared and analyzed with each batch of project samples. The total number of method blank sample analyses will be determined by the laboratory analytical batch size.

- Standard Reference Materials (SRM) will be used for each analysis. Sources of SRM include the U.S. EPA, commercially available material from CRADA certified vendors and/or laboratory produced solutions. SRM, when available and appropriate, will be processed and analyzed on a frequency of one per set of samples.

- Completeness is the evaluation of the amount of valid data generated versus the total set of data produced from a particular sampling and analysis event. Valid data is determined by independent confirmation of compliance with method-specific and project-specific data quality objectives. Laboratory data completeness will be calculated using an approach similar to field data completeness (Section 12.1) using the following equation:

$$\frac{Number\ of\ Valid\ Sample\ Results}{Total\ Number\ of\ Samples\ Planned}\ X\ 100 = \%\ Complete$$

13. CORRECTIVE ACTION

13.1 Field Corrective Action

The Project Manager and Field QA Officer will be responsible for ensuring the quality of the sampling procedures and environmental data and as such, will be responsible for initiating corrective action when appropriate. Corrective action is intended to address unacceptable procedures or deficient quality control performance.

The corrective action procedures will be as follows:

- Identify/define the problem.
- Assign responsibility for investigating the problem.
- Investigate/determine the cause of the problem.
- Determine an appropriate corrective action to eliminate the problem.
- Implement the corrective action.
- Evaluate the effectiveness of the corrective action.
- Verify that the corrective action has eliminated the problem.
- Prepare a written record detailing the problem, corrective action utilized and solution of the problem.
- Submit the Corrective Action Record (CAR) to the Project Manager who initiated the corrective action and the Project QA Officer.

The above procedures may be implemented through the use of the System Audits as previously described in Section 10. Any Field Team member of the project may initiate corrective action procedures by reporting in writing the nature of the suspected problem to the Project Manager or Project QA Officer. The Project Manager will begin corrective action by relating the problem to appropriate personnel.

13.2 Laboratory Corrective Action

The following paragraphs define the corrective action decision process relative to possible non-compliant events encountered during laboratory analysis of the project samples. Corrective actions will be initiated by the laboratory QA personnel and will be implemented by laboratory staff chemists under the oversight of the laboratory QA personnel. As with field corrective actions, the laboratory QA personnel will document the problem, the corrective action undertaken and the resolution of the problem. The corrective actions will be performed prior to release of the data from the laboratory.

Documentation will be provided to the Laboratory QA Officer, Haley & Aldrich QAO, Haley & Aldrich Project Manager and MLC Project Manager.

13.3 Corrective Action During Data Validation and Data Assessment

The Haley & Aldrich QAO may identify the need for corrective action during either the data validation or data assessment processes. Potential types of corrective action may include re-sampling by the field team or re-injection/re-analysis of samples by the laboratory (if possible).

When the Haley & Aldrich QAO identifies a situation requiring corrective action, MLC's Project Manager will be responsible for approving the corrective action, including re-sampling, during data assessment.

All corrective actions will be documented by the Haley & Aldrich Project Manager.

14. QUALITY ASSURANCE (QA) REPORTS

Critically important to the successful implementation of the QA Plan is a reporting system that provides the means by which the program can be reviewed, problems identified and programmatic changes made to improve the plan.

QA reports to management include:

- Audit reports summarizing internal and external audits and responses to those audits;
- Results of performance evaluations conducted by internal and external teams;
- Daily QA/QC exception reports and corrective actions

QA/QC corrective action reports will be prepared by the Haley & Aldrich QAO when appropriate. These reports will assist project and/or laboratory management in identifying problems and in making procedural changes, if warranted. The updated trend/QA charts prepared by the laboratory QA personnel will be distributed and reviewed by various levels of the laboratory management.

REFERENCES

1. United States Environmental Protection Agency, November 1999, "EPA Requirements for Quality Assurance Project Plans for Environmental Data Operations," EPA QA/R-5 Interim Final.

2. United States Environmental Protection Agency, April 1998, "U.S. EPA RCRA QAPP Instructions," U.S. EPA Region 5, Revision.

3. United States Environmental Protection Agency, February 1991, "Preparation Aids for the Development of Category I Quality Assurance Project Plans," U.S. EPA/600/8-91/003, Risk Reduction Engineering Laboratory, Office of Research and Development, Cincinnati, Ohio.

4. United States Environmental Protection Agency, September 1993, "Data Quality Objectives Process for Superfund Interim Final Guidance," U.S. EPA/540/R-93-071, Office of Solid Waste and Emergency Response (OSWER).

5. United States Environmental Protection Agency, April 1992, "Specifications and Guidance for Contaminant-Free Sample Containers," OSWER Directive 9240.0-05A.

6. United States Environmental Protection Agency, "U.S. EPA Contract Laboratory Program National Functional Guidelines for Organic Data Review," EPA-540-R-08-01, June 2008.

7. United States Environmental Protection Agency, "U.S. EPA Contract Laboratory Program National Functional Guidelines for Inorganic Data Review," EPA-540-R-04-004, October 2004.

8. United States Environmental Protection Agency, November 1986, "Test Methods for Evaluating Solid Waste, Office of Solid Waste," U.S. EPA, SW-846, with updates.

9. Ohio Environmental Protection Agency, September 1998, "Guidelines and Specifications for Preparing Quality Assurance Project Plans," Department of Emergency and Remedial Response.

TABLES

TABLE 1
PARAMETER LIST, ANALYTICAL METHODS AND
TARGET QUANTITATION LIMITS
TEXTILEATHER CORRECTIVE ACTION
TOLEDO, OHIO

	CAS #	Analytical Method		Detection Limits Limits[1]			
		Water	Soil	Lab RL Water (µg/L)	Target MDL Water (µg/L)	Lab RL Soil (µg/kg)	Target MDL Soil (µg/kg)
TCL Volatile Organic Compounds (VOC)							
Acetone	67-64-1	SW8260	SW8260	50	25	50.0	25
Benzene	71-43-2	SW8260	SW8260	1	0.27	2.0	1.1
Bromochloromethane	74-97-5	SW8260	SW8260	1	0.44	2.0	0.78
Bromodichloromethane	75-27-4	SW8260	SW8260	1	0.37	2.0	0.46
Bromoform	75-25-2	SW8260	SW8260	1	0.22	2.0	0.65
Bromomethane	74-83-9	SW8260	SW8260	1	0.47	2.0	0.88
2-Butanone	78-93-3	SW8260	SW8260	50	1.7	50.0	7.1
Carbon disulfide	75-15-0	SW8260	SW8260	1	0.29	5.0	0.67
Carbon tetrachloride	56-23-5	SW8260	SW8260	1	0.27	2.0	0.67
Chlorobenzene	108-90-7	SW8260	SW8260	1	0.38	2.0	0.72
Chloroethane	75-00-3	SW8260	SW8260	1	0.41	5.0	1
Chloroform	67-66-3	SW8260	SW8260	1	0.47	2.0	0.48
Chloromethane	74-87-3	SW8260	SW8260	1	0.2	2.0	1.1
Cyclohexane	110-82-7	SW8260	SW8260	5	0.2	10.0	0.57
Dibromochloromethane	124-48-1	SW8260	SW8260	1	0.3	2.0	0.64
1,2-Dibromo-3-chloropropane	96-12-8	SW8260	SW8260	5	0.2	5.0	1.5
1,2-Dibromoethane	106-93-4	SW8260	SW8260	1	0.34	2.0	0.67
1,2-Dichlorobenzene	95-50-1	SW8260	SW8260	1	0.2	2.0	0.67
1,3-Dichlorobenzene	541-73-1	SW8260	SW8260	1	0.49	2.0	1.5
1,4-Dichlorobenzene	106-46-7	SW8260	SW8260	1	0.25	2.0	0.72
Dichlorodifluoromethane	75-71-8	SW8260	SW8260	1	0.14	2.0	1.4
1,1-Dichloroethane	75-34-3	SW8260	SW8260	1	0.32	2.0	0.67
1,2-Dichloroethane	107-06-2	SW8260	SW8260	1	0.38	2.0	0.51
1,1-Dichloroethene	75-35-4	SW8260	SW8260	1	0.36	2.0	0.67
cis-1,2-Dichloroethene	156-59-2	SW8260	SW8260	1	0.38	2.0	0.67
trans-1,2-Dichloroethene	156-60-5	SW8260	SW8260	1	0.36	2.0	0.62
1,2-Dichloropropane	78-87-5	SW8260	SW8260	1	0.33	2.0	0.67
cis-1,3-Dichloropropene	10061-01-5	SW8260	SW8260	1	0.19	2.0	0.35
trans-1,3-Dichloropropene	10061-02-6	SW8260	SW8260	1	0.16	2.0	0.55
Ethylbenzene	100-41-4	SW8260	SW8260	1	0.32	2.0	0.98
2-Hexanone	561-78-6	SW8260	SW8260	50	2.2	50.0	8.5
Isopropylbenzene	98-82-8	SW8260	SW8260	1	0.24	2.0	0.67
Mehtyl acetate	79-20-9	SW8260	SW8260	10	0.28	10.0	1.6
Methylene chloride	75-09-02	SW8260	SW8260	5	0.15	10.0	1.6
Methylcyclohexane	108-87-2	SW8260	SW8260	5	0.21	10.0	3.3
4-Methly-2-pentanone	108-10-1	SW8260	SW8260	10	1.6	50.0	3.4
Methyl tert-butyl ether	1634-04-4	SW8260	SW8260	1	0.32	2.0	0.67
Propyl benzene,n-	103-65-1	SW8260	SW8260	1	0.22	2.0	0.86
Styrene	100-42-5	SW8260	SW8260	1	0.22	2.0	0.74
1,1,2,2-Tetrachloroethane	79-34-5	SW8260	SW8260	1	0.25	2.0	0.89
Tetrachloroethene	127-18-4	SW8260	SW8260	1	0.22	2.0	0.67
Tetrahydrofuran	109-99-9	SW8260	SW8260	20	3.3	20.0	8.4
Toluene	108-88-3	SW8260	SW8260	1	0.33	2.0	0.89
1,2,4-Trichlorobenzene	120-82-1	SW8260	SW8260	1	0.24	2.0	0.67
1,1,1-Trichloroethane	71-55-6	SW8260	SW8260	1	0.35	2.0	0.85
1,1,2-Trichloroethane	79-00-5	SW8260	SW8260	1	0.38	5.0	1.19
Trichloroethene	79-01-6	SW8260	SW8260	1	0.37	2.0	1
Trichlorofluoromethane	75-69-4	SW8260	SW8260	1	0.3	2.0	0.67
1,1,2-Trichloro-1,2,2-trifluoroeth	76-13-1	SW8260	SW8260	1	0.26	2.0	0.76
Vinyl Chloride	75-01-4	SW8260	SW8260	1	0.36	2.0	1.01
Xylenes (total)	1330-20-7	SW8260	SW8260	3	0.87	5.0	1.9

TABLE 1
PARAMETER LIST, ANALYTICAL METHODS AND
TARGET QUANTITATION LIMITS
TEXTILEATHER CORRECTIVE ACTION
TOLEDO, OHIO

	CAS #	Analytical Method		Detection Limits Limits[1]			
		Water	Soil	Lab RL Water (µg/L)	Target MDL Water (µg/L)	Lab RL Soil (µg/kg)	Target MDL Soil (µg/kg)
TCL Semi-Volatile Organic Compounds (SVOC)							
Acenapthene	83-32-9	SW8270	SW8270	2.000	0.5	330.0	14
Acenaphthylene	208-96-8	SW8270	SW8270	2.000	0.4	330.0	20
Acetophenone	98-86-2	SW8270	SW8270	10.000	8	330.0	35
Anthracene	120-12-7	SW8270	SW8270	2.000	0.2	330.0	9
Atrazine	1912-24-9	SW8270	SW8270	10.000	9.8	330.0	73
Benzaldehyde	100-52-7	SW8270	SW8270	10.000	8.4	1670.0	297
Benzo(a)anthracene	56-55-3	SW8270	SW8270	2.000	0.7	330.0	11
Benzo(b)fluoranthene	205-99-2	SW8270	SW8270	2.000	0.7	330.0	38
Benoz(k)fluorathene	207-08-9	SW8270	SW8270	2.000	0.8	330.0	37
Benzo(g,h,I)perylene	191-24-2	SW8270	SW8270	2.000	0.5	330.0	9
Benzo(a)pyrene	50-32-8	SW8270	SW8270	2.000	0.6	330.0	8
1,1-Biphenyl	92-52-4	SW8270	SW8270	10.000	8.2	330.0	104
4-Bromophenylphenyl ether	101-55-3	SW8270	SW8270	10.000	3	330.0	150
Butyl benzyl phthalate	85-68-7	SW8270	SW8270	10.000	3.7	330.0	125
di-n-Butyl phthalate	87-74-2	SW8270	SW8270	10.000	3.2	330.0	141
Caprolactam	105-60-2	SW8270	SW8270	10.000	2.8	330.0	102
Carbazole	86-74-8	SW8270	SW8270	10.000	0.4	330.0	16
4-Chloroaniline	106-47-8	SW8270	SW8270	10.000	4.8	330.0	103
bis(2-Chloroethoxy)methane	111-91-1	SW8270	SW8270	10.000	2.6	330.0	128
bis(2-Chloroethyl)ether	111-44-4	SW8270	SW8270	10.000	4.1	330.0	151
bis(2-Chloroisopropyl)ether	108-60-1	SW8270	SW8270	10.000	3.5	330.0	158
4-Chloro-3-methylphenol	59-50-7	SW8270	SW8270	10.000	2.8	330.0	142
2-Chloronaphthalene	91-58-7	SW8270	SW8270	10.000	2.9	330.0	150
2-Chlorophenol	95-57-8	SW8270	SW8270	10.000	2.4	330.0	149
4-Chlorophenyl phenyl ether	7005-72-3	SW8270	SW8270	10.000	2.8	330.0	148
Chrysene	218-01-9	SW8270	SW8270	2.000	0.5	330.0	31
Dibenz(a,h)anthracene	53-70-3	SW8270	SW8270	2.000	0.6	330.0	15
Dibenzofuran	132-64-9	SW8270	SW8270	10.000	0.7	330.0	14
3,3-Dichlorobenzidine	91-94-1	SW8270	SW8270	10.000	6.4	660.0	48
2,4-Dichlorophenol	120-83-2	SW8270	SW8270	10.000	2.3	330.0	131
Diethylphthalate	84-66-2	SW8270	SW8270	10.000	2.8	330.0	148
2,4-Dimethylphenol	105-67-9	SW8270	SW8270	10.000	2.6	330.0	137
Dimethylphthalate	121-11-3	SW8270	SW8270	10.000	2.7	330.0	157
4,6-Dinitro-2-methylphenol	534-52-1	SW8270	SW8270	25.000	3.3	830.0	112
2,4-Dinitrophenol	51-28-5	SW8270	SW8270	25.000	3.1	830.0	99
2,4-Dinitrotoluene	121-14-2	SW8270	SW8270	10.000	3	330.0	140
2,6-Dinitrotoluene	606-20-2	SW8270	SW8270	10.000	3	330.0	150
bis(2-Ethylhexyl)phthalate	117-81-7	SW8270	SW8270	10.000	3.3	330.0	126
Fluoranthene	206-44-0	SW8270	SW8270	2.000	0.7	330.0	11
Fluorene	86-73-7	SW8270	SW8270	2.000	0.7	330.0	20
Hexachlorobenzene	118-74-1	SW8270	SW8270	10.000	2.7	330.0	153
Hexachlorobutadiene	87-68-3	SW8270	SW8270	10.000	2.7	330.0	174
Hexachlorocyclopentadiene	77-47-4	SW8270	SW8270	10.000	1.9	330.0	101
Hexachloroethane	67-72-1	SW8270	SW8270	10.000	2.4	330.0	150
Indeno(1,2,3-cd)pyrene	193-39-5	SW8270	SW8270	2.000	0.5	330.0	31
Isophorone	78-59-1	SW8270	SW8270	10.000	2.8	330.0	135
2-Methylnaphthalene	91-57-6	SW8270	SW8270	2.000	0.6	330.0	21
2-Methylphenol	95-48-7	SW8270	SW8270	10.000	2.6	330.0	189
3/4-Methylphenol	106-44-5	SW8270	SW8270	10.000	2.3	330.0	166
Naphthalene	91-20-3	SW8270	SW8270	2.000	0.5	330.0	14
2-Nitroaniline	88-74-4	SW8270	SW8270	25.000	2.3	830.0	121

TABLE 1
PARAMETER LIST, ANALYTICAL METHODS AND
TARGET QUANTITATION LIMITS
TEXTILEATHER CORRECTIVE ACTION
TOLEDO, OHIO

	CAS #	Analytical Method		Detection Limits Limits[1]			
		Water	Soil	Lab RL Water (µg/L)	Target MDL Water (µg/L)	Lab RL Soil (µg/kg)	Target MDL Soil (µg/kg)
TCL Semi-Volatile Organic Compounds (SVOC) (continued)							
3-Nitroaniline	99-09-2	SW8270	SW8270	25.000	2.9	830.0	273
4-Nitroaniline	100-01-6	SW8270	SW8270	25.000	3.4	830.0	252
Nitrobenzene	98-95-3	SW8270	SW8270	10.000	2.5	330.0	159
2-Nitrophenol	88-75-5	SW8270	SW8270	10.000	3	330.0	196
4-Nitrophenol	100-02-7	SW8270	SW8270	25.000	5.3	830.0	153
N-Nitroso-di-n-propylamine	621-64-7	SW8270	SW8270	10.000	2.7	330.0	152
N-Nitrosodiphenylamine (diphen	86-30-6	SW8270	SW8270	10.000	4	330.0	183
di-n-Octyl phthalate	117-84-0	SW8270	SW8270	10.000	3.7	330.0	135
Pentachlorophenol	87-86-5	SW8270	SW8270	25.000	2.6	830.0	123
Phenanthrene	85-01-8	SW8270	SW8270	2.000	0.7	330.0	10
Phenol	108-95-2	SW8270	SW8270	10.000	1.7	330.0	131
Pyrene	129-00-0	SW8270	SW8270	2.000	0.6	330.0	23
2,4,5-Trichlorophenol	95-95-4	SW8270	SW8270	25.000	2.7	830.0	148
2,4,6-Trichlorophenol	88-06-2	SW8270	SW8270	10.000	3	330.0	149
TCL Polychlorinated Biphenyls (PCB)							
Aroclor 1016	12674-11-2	SW8082	SW8082	0.5	0.132	33	21
Aroclor 1221	11104-28-2	SW8082	SW8082	0.5	0.186	33	11
Aroclor 1232	11141-16-5	SW8082	SW8082	0.5	0.215	33	16
Aroclor 1242	53469-21-9	SW8082	SW8082	0.5	0.188	33	26
Aroclor 1248	12672-29-6	SW8082	SW8082	0.5	0.141	33	30
Aroclor 1254	11097-69-1	SW8082	SW8082	0.5	0.111	33	11
Aroclor 1260	11096-82-5	SW8082	SW8082	0.5	0.2	33	28
Glycols							
Ethylene glycol	107-21-1	SW8015	SW8015	10 mg/l	.5 mg/l	10 mg/kg	1.4 mg/kg
TAL Metals (ICP and CVAA)							
Antimony	7440-36-0	SW6010	SW6010	10	5.9	10000	800
Arsenic	7440-38-2	SW6010	SW6010	10	4	1000	700
Barium	7440-39-3	SW6010	SW6010	10	6	2000	700
Beryllium	7440-41-7	SW6010	SW6010	4	1	1000	100
Cadmium	7440-43-9	SW6010	SW6010	1	0.6	1000	200
Chromium	7440-47-3	SW6010	SW6010	5	1	1000	200
Cobalt	7440-48-4	SW6010	SW6010	20	2	1000	300
Copper	7440-50-8	SW6010	SW6010	10	3	2000	500
Lead	7439-92-1	SW6010	SW6010	5	2.9	1000	600
Manganese	7439-96-5	SW6010	SW6010	15	1.5	1000	200
Mercury	7439-97-6	SW6010	SW6010	0.2	0.1	100	7.7
Nickel	7440-02-0	SW6010	SW6010	10	2	1000	400
Selenium	7782-49-2	SW6010	SW6010	10	3.9	2000	600
Silver	7740-22-4	SW6010	SW6010	5	2	1000	200
Thallium	7440-28-0	SW6010	SW6010	10	8.4	2000	900
Vanadium	7440-62-2	SW6010	SW6010	20	7	10000	900
Zinc	7440-66-6	SW6010	SW6010	50	8	10000	500
Cyanide (total)	57-12-5	SW6010	SW6010	5	3	2000	1800

TABLE 1
PARAMETER LIST, ANALYTICAL METHODS AND
TARGET QUANTITATION LIMITS
TEXTILEATHER CORRECTIVE ACTION
TOLEDO, OHIO

	CAS #	Analytical Method		Detection Limits Limits[1]			
		Water	Soil	Lab RL Water (µg/L)	Target MDL Water (µg/L)	Lab RL Soil (µg/kg)	Target MDL Soil (µg/kg)
TAL Metals (ICPMS)							
Antimony	7440-36-0	SW6020	SW6020	2	0.07		
Arsenic	7440-38-2	SW6020	SW6020	2	0.06		
Barium	7440-39-3	SW6020	SW6020	2	0.07		
Beryllium	7440-41-7	SW6020	SW6020	2	0.08		
Cadmium	7440-43-9	SW6020	SW6020	1	0.03		
Chromium	7440-47-3	SW6020	SW6020	2	0.3		
Cobalt	7440-48-4	SW6020	SW6020	2	0.2		
Copper	7440-50-8	SW6020	SW6020	5	0.3		
Lead	7439-92-1	SW6020	SW6020	2	0.09		
Manganese	7439-96-5	SW6020	SW6020	5	0.2		
Nickel	7440-02-0	SW6020	SW6020	5	0.09		
Selenium	7782-49-2	SW6020	SW6020	2	0.07		
Silver	7740-22-4	SW6020	SW6020	2	0.3		
Thallium	7440-28-0	SW6020	SW6020	2	0.05		
Vanadium	7440-62-2	SW6020	SW6020	4	0.2		
Zinc	7440-66-6	SW6020	SW6020	50	0.9		

Notes:

1. Please note that target these quantitation limits are presented for guidance only. Actual quantitation limits are highly matrix dependent and may be elevated due to matrix effects, QA/QC problems and high concentrations of target and non-target analytes.
   Lab MDL - Laboratory Method Detection Limit
   PQL - Target Practical Quantitation Limit

2. TAL Metals aluminum, calcium, iron, magnesium, potassium and sodium will not be analyzed due to the low risk associated with these parameters.

3. TCL VOC and TCL SVOC analysis excludes dioxins, pesticides, and herbicides.

TABLE 2
SAMPLE CONTAINER, PRESERVATION, SHIPPING AND PACKAGING REQUIREMENTS
TEXTILEATHER CORRECTIVE ACTION
TOLEDO, OHIO

Analyses [1]	*Sample Containers* [2]	*Preservation*	*Maximum Holding Time From Sample Collection* [3]	*Volume of Sample*	*Shipping*	*Normal Packaging*
Soil						
Volatile Organic Compounds	Three 5g EnCore Sampler ™ per analysis[4] (or equivalent)	Iced, </= 4 +/- 2 C	48 hours for extraction 14 days after extraction for analysis	Fill completely	Overnight or hand deliver	Foil Pouch
Semi Volatile Organic Compounds and Polychlorinated biphenyls	One 8-ounce glass jar per analysis	Iced, </= 4 +/- 2 C	14 days for extraction 40 days after	Fill to shoulder of jar	Overnight or hand deliver	Bubble Pack or Foam Chips
Target Analyte List Metals	One 4-ounce glass jar per analysis	Iced, </= 4 +/- 2 C	180 days (mercury 28 days) for analysis	Fill to shoulder of jar	Overnight or hand deliver	Bubble Pack or Foam Chips
Total Cyanide	One 4-ounce glass jar	Iced, </= 4 +/- 2 C	14 days for analysis	Fill to shoulder of jar	Overnight or hand deliver	Bubble Pack or Foam Chips
Chromium VI	One 4-ounce glass jar	Iced, </= 4 +/- 2 C	24 hours for analysis	Fill to shoulder of jar	Overnight or hand deliver	Bubble Pack or Foam Chips

Notes:

[1] Analyses-sample containers, preservation, shipping and packaging requirement are applicable to TCL/TAL parameter groups.

[2] Multiple analysess for a soil sample may not require additional containers for each parameter.

[3] The technical holding times are based on time elapsed from sample collection.

[4] If VOCs are the only parameter analyzed, one 4-ounce plastic container will be collected for dry weight determination.

TABLE 2
SAMPLE CONTAINER, PRESERVATION, SHIPPING AND PACKAGING REQUIREMENTS
TEXTILEATHER CORRECTIVE ACTION
TOLEDO, OHIO

Analysis [1]	*Sample Containers* [2]	*Preservation*	*Maximum Holding Time From Sample Collection* [3]	*Volume of Sample*	*Shipping*	*Normal Packaging*
Groundwater						
Volatile Organic Compounds	Three 40 mL teflon-lined septum vials per analysis	HCL to pH <2 Iced, </= 4 +/- 2 C	14 days for analysis	Fill completely, no air bubbles	Overnight or hand deliver	Foam Liner or Bubble Pack
Semi Volatile Organic Compounds and Polychlorinated biphenyls	Two 1 liter pamber glass bottles per analysis	Iced, </= 4 +/- 2 C	7 days for extraction 40 days after extraction for analysis	Fill to neck of bottle	Overnight or hand deliver	Bubble Pack or Foam Chips
Target Analyte List Metals	One 1 liter plastic bottle	HNO_3 to pH <2, Iced, </= 4 +/- 2 C	180 days (mercury 28 days) for analysis	Fill to neck of bottle	Overnight or hand deliver	Bubble Pack or Foam Chips
Total Cyanide	One 500 ml plastic bottle	NaOH to pH>12 Iced, </= 4 +/- 2 C	14 days for analysis	Fill to neck of bottle	Overnight or hand deliver	Bubble Pack or Foam Chips
Chromium VI	One 250 ml plastic bottle	Iced, </= 4 +/- 2 C	24 hours for analysis	Fill to neck of bottle	Overnight or hand deliver	Bubble Pack or Foam Chips

APPENDIX A

Laboratory Quality Assurance Manual



Quality Assurance Manual

TestAmerica Nashville

2960 Foster Creighton Drive
Nashville, TN 37204
Phone: 615-726-0177/ 800-765-0980
Fax: 615-726-3404

www.testamericainc.com

Effective Date of Revision 20: April 6, 2010

Vision, Mission & Core Values

Vision

TestAmerica will be the industry leader for environmental testing and data deliverables.

Mission Statement

TestAmerica is committed to providing exceptional client service, highest quality legally defensible data, and the most comprehensive range of capabilities in the environmental testing industry.

Core Values

Integrity: We adhere to the highest moral and ethical principles in all that we do.

Client Service: We ensure our clients' satisfaction and success.

Performance: We set challenging goals, hold one another accountable, and reward results.

Technical Excellence: We continually invest in new technologies to provide exceptional data quality and credibility.

Teamwork: We help each other succeed through a cooperative team effort, in an atmosphere of civility and respect.

People: We invest in the long term professional development and success of our employees.

Growth: We manage our business for profitable growth.

TestAmerica

THE LEADER IN ENVIRONMENTAL TESTING

Quality Assurance Manual

TestAmerica Nashville
2960 Foster Creighton Drive
Nashville, TN 37204
Phone: 615-726-0177/ 800-765-0980
Fax: 615-726-3404
www.testamericainc.com

Effective Date of Revision 20: April 6, 2010

Approval Signatures

Laboratory Director – Johnny A. Mitchell — Date: 3.23.10

Quality Assurance Manager - Eric S. Smith — Date: 3-23-10

Technical Director – Michael H. Dunn — Date: 3-23-10

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REFERENCED CORPORATE SOPs AND POLICIES

SOP/Policy Reference	Title
CA-Q-S-001	Solvent and Acid Lot Testing and Approval
CA-Q-S-002	Acceptable Manual Integration Practices
CA-Q-S-004	Internal Auditing
CA-Q-S-005	Calibration Curves (General)
CA-Q-S-008	Management Systems Review
CW-Q-S-001	Corporate Document Control and Archiving
CW-Q-S-002	Writing a Standard Operating Procedure (SOPs)
CA-L-S-001	Internal Investigation of Potential Data Discrepancies and Determination for Data Recall
CA-L-S-002	Subcontracting Procedures
CA-L-P-002	Ethics Policy
CA-L-P-002	Contract Compliance Policy
CW-F-P-002	Authorization Matrix
CW-F-P-004	Procurement and Contracts Policy
CA-C-S-001	Work Sharing Process
CA-T-P-001	Qualified Products List
CW-F-S-004	Controlled Purchases Policy

SECTION 3

INTRODUCTION
(NELAC 5.1 - 5.3)

3.1 INTRODUCTION AND COMPLIANCE REFERENCES

TestAmerica Nashville's Quality Assurance Manual (QAM) is a document prepared to define the overall policies, organization objectives and functional responsibilities for achieving TestAmerica's data quality goals. Each TestAmerica laboratory maintains a local perspective in its scope of services and client relations and maintains a national perspective in terms of quality.

The QAM has been prepared to assure compliance with the 2003 National Environmental Laboratory Accreditation Conference (NELAC) standards and ISO/IEC Guide 17025 (2005). In addition, the policies and procedures outlined in this manual are compliant with TestAmerica's Corporate Quality Management Plan (CQMP) and the various accreditation and certification programs listed in Appendix 6. The CQMP provides a summary of TestAmerica's quality and data integrity system. It contains requirements and general guidelines under which all TestAmerica facilities shall conduct their operations.

The QAM has been prepared to be consistent with the requirements of the following documents:

- EPA 600/4-88/039, *Methods for the Determination of Organic Compounds in Drinking Water,* EPA, Revised July 1991.
- EPA 600/R-95/131, *Methods for the Determination of Organic Compounds in Drinking Water,* Supplement III, EPA, August 1995.
- EPA 600/4-79-019, *Handbook for Analytical Quality Control in Water and Wastewater Laboratories,* EPA, March 1979.
- EPA SW-846, *Test Methods for Evaluating Solid Waste Physical/Chemical Methods (SW846)*, Third Edition, September 1986, Final Update 1, July 1992; Final Update IIA, August 1993; Final Update II, September 1994; Final Update IIB, January 1995; Final Update III, December 1996; Final Update IIIB, June 2005, and Final Update IV, January 2008.
- Federal Register, 40 CFR Parts 136, 141, 172, 173, 178, 179 and 261.
- USEPA Contract Laboratory Program, *Statement of Work for Inorganics & Organics Analysis, Multi-Media, Multi-Concentration,* SOM and ISM, current revisions.
- APHA, *Standard Methods for the Examination of Water and Wastewater,* 18th Edition, 19th, 20th and 21st Edition.
- *U.S. Department of Energy* Order 414.1B, *Quality Assurance*, Approved April 29, 2004.
- *US Department of Energy* Order 414.1 C, *Quality Assurance,* June 17, 2005.
- *U.S. Department of Energy*, *Quality Systems for Analytical Services*, Revision 2.4, October 28, 2008.
- U.S. Department of Defense, *Quality Systems Manual for Environmental Laboratories*, Final Version 3, January 2006.
- U.S. Department of Defense, *Air Force Center for Environmental Excellence Quality Assurance Project Plan (QAPP),* Version 4.0.02, May 2006.

3.2 TERMS AND DEFINITIONS

A Quality Assurance Program is a company-wide system designed to ensure that data produced by TestAmerica Nashville conforms to the standards set by state and/or federal regulations. The program functions at the management level through company goals and management policies, and at the analytical level through Standard Operating Procedures (SOPs) and quality control. The TestAmerica program is designed to minimize systematic error, encourage constructive, documented problem solving, and provide a framework for continuous improvement within the organization.

Refer to Appendix 5 for the Glossary/Acronyms.

3.3 SCOPE / FIELDS OF TESTING

TestAmerica Nashville analyzes a broad range of environmental and industrial samples every month. Sample matrices vary among air, drinking water, effluent water, groundwater, hazardous waste, sludge and soils. The Quality Assurance Program contains specific procedures and methods to test samples of differing matrices for chemical and physical parameters. The QA Program also contains guidelines on maintaining documentation of analytical process, reviewing results, servicing clients and tracking samples through the laboratory. The technical and service requirements of all requests to provide analyses are thoroughly evaluated before commitments are made to accept the work. Measurements are made using published reference methods or methods developed and validated by the laboratory.

The methods covered by this manual include the most frequently requested water, air, industrial waste, and soil methodologies needed to provide analytical services in the United States and its territories. The specific list of test methods used by the laboratory can be found in the Control Limits Manual. The approach of this manual is to define the minimum level of quality assurance and quality control necessary to meet requirements. All methods performed by TestAmerica Nashville meet these criteria, as appropriate. In some instances, quality assurance project plans (QAPPs), project specific data quality objectives (DQOs) or local regulations may require criteria other than those contained in this manual. In these cases, the laboratory abides by the requested criteria following review and acceptance of the requirements by the Laboratory Director, the Technical Director, and the Quality Assurance Manager. In some cases, QAPPs and DQOs may specify less stringent requirements. The Laboratory Director, the Technical Director, and the QA Manager must determine if it is in the lab's best interest to follow the less stringent requirements.

3.4 MANAGEMENT OF THE MANUAL

3.4.1 Review Process

This manual is reviewed annually by senior laboratory management to assure that it reflects current practices and meets the requirements of TestAmerica Nashville's clients and regulators as well as the CQMP. Occasionally, the manual may need changes in order to meet new or changing regulations and operations. The QA Manager reviews the changes in the normal course of business and incorporate changes into revised sections of the document. The updates are reviewed by the QA Manager, Laboratory Director, Technical Director, relevant operational staff and Corporate Quality Assurance (if a change is made to the Corporate template) and then incorporated into the document in periodic updates. The QAM is based on a Corporate QAM

Template that is prepared and reviewed annually by the Corporate Quality Department. Necessary changes are coordinated by the Corporate Quality Department and distributed electronically to each laboratory for inclusion in the laboratory specific QA Manuals.

Policies in the QAM that require immediate attention are addressed through the use of a Laboratory QA/QC Policy Memoranda. QA/QC Policy Memoranda are published from time to time to facilitate immediate changes to QA/QC Policy. QA/QC Policy Memoranda supersede the QAM and all other SOPs (refer to Section 5.3). All policy memoranda are dated, archived and distributed by their placement into the front of the QAM between the signature page and Section 2. At a minimum, each policy memorandum is approved by the same authorized signatories as shown on the cover page of the QA Manual. The QA/QC Policy Memoranda are incorporated into the QAM during the periodic updates. Policy memorandum may also include an expiration date if appropriate. An example format can be found in Figure 3-1.

Laboratory-specific QAM changes are approved and documented through the Management of Change process. (Refer to SOP NV08-161, Management of Change System.)

3.4.2 Control

Copyright information pertaining to this QA Manual is located on the Cover Page of this manual. The procedure for control of distribution of the QA manual is incorporated by reference to laboratory SOP NV08-152: Document Control.

Figure 3-1.

Laboratory QA/QC Policy Memorandum # _____

Effective Date: _____ Expiration Date: When Appropriate QAM Section is Revised

_____	_____
Technical Director Approval Date	Quality Assurance Approval Date
_____	
Laboratory Director Approval Date	

1. **Purpose**

2. **Procedure**

3. **Documentation**

4. **Attachments**

5. **References/Cross References**

SECTION 4

ORGANIZATION AND MANAGEMENT
(NELAC 5.4.1)

4.1 OVERVIEW

TestAmerica Nashville is a local operating unit of TestAmerica Laboratories, Inc. This Quality Assurance Manual (QAM) is applicable to the TestAmerica Nashville laboratory only.

TestAmerica Nashville
2960 Foster Creighton Drive
Nashville, TN 37204

EPA ID: TN00032

The organizational structure, responsibilities, and authorities of the corporate staff of TestAmerica Laboratories, Inc are presented in the CQMP. The laboratory has day-to-day independent operational authority overseen by corporate officers (e.g., President, Chief Operating Officer, General Managers, VP of Client and Technical Services, Corporate Director of Technical Services, Legal and Contracts Director, National Accounts Director, Ethics and Compliance Directors, Director of Quality and Client Advocacy, Corporate Quality Directors, Corporate Quality Assurance, etc.). The laboratory operational and support staff work under direction of the Laboratory Director. The organizational structure for Corporate is presented in Figure 4-1 and the laboratory's organization chart can be found in Appendix 2.

4.2 ROLES AND RESPONSIBILITIES

In order for the Quality Assurance Program to function properly, all members of the staff must clearly understand and meet their individual responsibilities as they relate to the quality program. The following descriptions define each role in its relationship to the Quality Assurance Program. When necessary, more extensive job descriptions may be maintained by laboratory management.

4.2.1 Quality Assurance Program

The responsibility for quality lies with every employee of TestAmerica Nashville. All employees have access to the QAM, are trained to this manual, and are responsible for upholding the standards therein. Each person carries out his/her daily tasks in a manner consistent with the goals and in accordance with the procedures in this manual and the laboratory's SOPs. Role descriptions for Corporate personnel are defined in the CQMP. This manual is specific to the operations of TestAmerica's Nashville laboratory

4.2.2 Laboratory Director

TestAmerica Nashville's Laboratory Director is responsible for the overall quality, safety, financial, technical, human resource and service performance of the whole laboratory and reports to their respective General Manager. The Laboratory Director provides the resources

necessary to implement and maintain an effective and comprehensive Quality Assurance and Data Integrity Program.

Specific responsibilities include, but are not limited to:

- Provides one or more technical directors for the appropriate fields of testing. The name(s) of the Technical Director is (are) included in the national database. If the Technical Director is absent for a period of time exceeding 15 consecutive calendar days, the Laboratory Director must designate another full time staff member meeting the qualifications of the Technical Director to temporarily perform this function. If the absence exceeds 65 consecutive calendar days, the primary accrediting authority must be notified in writing.
- Ensures that all analysts and supervisors have the appropriate education and training to properly carry out the duties assigned to them and ensures that this training has been documented.
- Ensures that personnel are free from any commercial, financial and other undue pressures which might adversely affect the quality of their work.
- Ensures TestAmerica's human resource policies are adhered to and maintained.
- Ensures that sufficient numbers of qualified personnel are employed to supervise and perform the work of the laboratory.
- Ensures that appropriate corrective actions are taken to address analyses identified as requiring such actions by internal and external performance or procedural audits. Procedures that do not meet the standards set forth in the QAM or laboratory SOPs may be temporarily suspended by the Laboratory Director. Once all corrective actions have been made, with the approval of the Quality Manager, the Laboratory Director has the responsibility to resume suspended/restricted work.
- Reviews and approves all SOPs prior to their implementation and ensures all approved SOPs are implemented and adhered to.
- Pursues and maintains appropriate laboratory certification and contract approvals. Supports ISO 17025 requirements.
- Ensures client specific reporting and quality control requirements are met.
- Captains the management team, consisting of the QA Manager, the Technical Director, and the Operations Manager(s) as direct reports.

4.2.3 Quality Assurance Manager

The QA Manager has responsibility and authority to ensure the continuous implementation of the quality system based on ISO 17025.

The QA Manager reports directly to the Laboratory Director and the Techical Director and has access to Corporate QA for advice and resources. This position is able to evaluate data objectively and perform assessments without outside (i. e., managerial) influence. Corporate QA may be used as a resource in dealing with regulatory requirements, certifications and other quality assurance related items. The QA Manager directs the activities of the QA officers to accomplish specific responsibilities, which include, but are not limited to:

- Having functions independent from laboratory operations for which he/she has quality assurance oversight.
- Maintaining and updating the QAM.
- Monitoring and evaluating laboratory certifications; scheduling proficiency testing samples.
- Monitoring and communicating regulatory changes that may affect the laboratory to management.
- Training and advising the laboratory staff on quality assurance/quality control procedures that are pertinent to their daily activities.
- Having a general knowledge of the analytical test methods for which data audit/review is performed (and/or having the means of getting this information when needed).
- Arranging for or conducting internal audits on quality systems and the technical operation.
- Maintains records of all ethics-related training, including the type and proof of attendance.
- Maintains, improves, and evaluates the corrective action database and the corrective and preventive action systems.
- Notifying laboratory management of deficiencies in the quality system and ensuring corrective action is taken. Procedures that do not meet the standards set forth in the QAM or laboratory SOPs are temporarily suspended following the procedures outlined in Section 13.
- Evaluation of the thoroughness and effectiveness of training.
- Monitoring standards of performance in quality control and quality assurance to ensure that systems are in place to produce the level of quality as defined in this document.
- Coordinating of document control of SOPs, MDLs, control limits, and miscellaneous forms and information.
- Reviews a percentage of all final data reports for internal consistency, including Chain of Custody (COC), correspondence with the analytical request, batch QC status, completeness of any corrective action statements, calculations, format, holding time, sensibility and completeness of the project file contents.
- Reviews of external audit reports and data validation requests.
- Follows up with audits to ensure client QAPP requirements are met.
- Ensuring communication with and preparation of various quality reports for the Laboratory Director, clients and/or Corporate QA.
- Development of suggestions and recommendations to improve quality systems.
- Research of current state and federal requirements and guidelines.
- Captains the QA team to enable communication and to distribute duties and responsibilities.

4.2.4 Technical Director

The Technical Director reports directly to the Laboratory Director. He/she is accountable for all analyses and analysts with respect to ISO 17025. The scope of responsibility ranges from the new-hire process and existing technology through the ongoing training and development

programs for existing analysts and second- and third-generation instrumentation. Specific responsibilities include, but are not limited to:

- Coordinating, writing, and reviewing preparation of all test methods, i. e., SOPs, with regard to quality, integrity, regulatory and optimum and efficient production techniques, and subsequent analyst training and interpretation of the SOPs for implementation and unusual project samples. He/she insures that the SOPs are properly managed and adhered to at the bench. He/she develops standard costing of SOPs to include supplies, labor, overhead, and capacity (design vs. demonstrated versus first-run yield) utilization.
- Reviewing and approving, with input from the QA Manager, proposals from marketing, in accordance with an established procedure for the review of requests and contracts. This procedure addresses the adequate definition of methods to be used for analysis and any limitations, the laboratory's capability and resources, the client's expectations. Differences are resolved before the contract is signed and work begins. A system documenting any significant changes is maintained, as well as pertinent discussions with the client regarding their requirements or the results of the analyses during the performance of the contract. All work subcontracted by the laboratory must be approved by the client. Any deviations from the contract must be disclosed to the client. Once the work has begun, any amendments to the contract must be discussed with the client and so documented.
- Monitoring the validity of the analyses performed and data generated in the laboratory. This activity begins with reviewing and supporting new business contracts, insuring data quality, analyzing internal and external non-conformances to identify root cause issues and implementing the resulting corrective and preventive actions, facilitating the data review process (training, development, and accountability at the bench), and providing technical and troubleshooting expertise on routine and unusual or complex problems.
- Providing training and development programs to applicable laboratory staff as new hires and, subsequently, on a scheduled basis. Training includes instruction on calculations, manual integration, and instrumentation management to include troubleshooting and preventive maintenance.
- Enhancing efficiency and improving quality through technical advances and improved LIMS utilization. Capital forecasting and instrument life cycle planning for second generation methods and instruments as well as asset inventory management.
- Coordinating sample management from "cradle to grave," insuring that no time is lost in locating samples.
- Scheduling all QA/QC-related requirements for compliance, e.g., MDLs, etc.
- Works with operations managers and department supervisors to communicate quality, technical, personnel, and instrumental issues for a consistent team approach.
- Coordinates audit responses with operations managers, department supervisors and QA Manager.

4.2.5 Analytical Operations Manager(s)

The Analytical Operations Managers manage and direct the analytical production sections of the laboratory. For Ohio VAP, this position is known as a Department Operations Manager. He/She

reports directly to the Laboratory Director. He/She assists the Technical Director in determining the most efficient instrument utilization. More specifically, he/she:

- Evaluates the level of internal/external non-conformances for all departments.
- Continuously evaluates production capacity and improves capacity utilization.
- Continuously evaluates turnaround time and addresses any problems that may hinder meeting the required and committed turnaround time from the various departments.
- Develops and improves the training of all analysts in cooperation with the Technical Director and QA Manager and in compliance with regulatory requirements.
- Ensures that scheduled instrument maintenance is completed.
- Is responsible for efficient utilization of supplies.
- Constantly monitors and modifies the processing of samples through the departments.
- Develops, reviews, and provides comment on technical SOPs.
- Works with department supervisors to ensure departmental compliance with the quality system.

4.2.6 Hazardous Waste Coordinator

The Hazardous Waste Coordinator reports directly to the Laboratory Director. The duties consist of:

- Staying current with the hazardous waste regulations.
- Continuing training on hazardous waste issues.
- Reviewing and updating annually the Hazardous Waste Contingency Plan in the Environmental Health & Safety Manual.
- Auditing the staff with regard to compliance with the Hazardous Waste Contingency Plan.
- Contacting the hazardous waste subcontractors for review of procedures and opportunities for minimization of waste.

4.2.7 Department Supervisors

Department supervisors report to the Operations Managers. Each one is responsible to:

- Ensure that analysts in their department adhere to applicable SOPs and the QA Manual. They perform frequent SOP and QA Manual review to determine if analysts are in compliance and if new, modified, and optimized measures are feasible and should be added to these documents.
- With regard to analysts, participates in the selection, training (as documented in Section 8.3), development of performance objectives and standards of performance, appraisal (measurement of objectives), scheduling, counseling, discipline, and motivation of analysts and documents these activities in accordance with systems developed by the QA and Personnel Departments. They evaluate staffing sufficiency and overtime needs. Training consists of familiarization with SOP, QC, Safety, and computer systems.

- Encourage the development of analysts to become cross-trained in various methods and/or operate multiple instruments efficiently while performing maintenance and documentation, self-supervise, and function as a department team.
- Provide guidance to analysts in resolving problems encountered daily during sample prep/analysis in conjunction with the Technical Director, Operations Manager, and/or QA Manager. Each is responsible for 100% of the data review and documentation, non-conformance and CAR issues, the timely and accurate completion of performance evaluation samples and MDLs, for his/her department.
- Ensure all logbooks are maintained, current, and properly labeled or archived.
- Report all non-conformance conditions to the QA Manager, Technical Director, Operations Managers, and/or Laboratory Director.
- Ensure that preventive maintenance is performed on instrumentation as detailed in the QA Manual or SOPs. He/she is responsible for developing and implementing a system for preventive maintenance, troubleshooting, and repairing or arranging for repair of instruments.
- Maintain adequate and valid inventory of reagents, standards, spare parts, and other relevant resources required to perform daily analysis.
- Achieve optimum turnaround time on analyses and compliance with holding times.
- Conduct efficiency and cost control evaluations on an ongoing basis to determine optimization of labor, supplies, overtime, first-run yield, capacity (designed vs. demonstrated), second- and third-generation production techniques/instruments, and long-term needs for budgetary planning.
- Develop, implement, and enhance calibration programs.
- Provide written responses to external and internal audit issues.

4.2.8 Laboratory Analysts

Laboratory analysts are responsible for conducting analysis and performing all tasks assigned to them by the group leader or department supervisor. The responsibilities of the analysts are listed below:

- Perform analyses by adhering to analytical and quality control protocols prescribed by current SOPs, this QA Manual, and project-specific plans honestly, accurately, timely, safely, and in the most cost-effective manner.
- Document standard and sample preparation, instrument calibration and maintenance, data calculations, sample matrix effects, and any observed non-conformance on worklists, benchsheets, lab notebooks and/or the Non-Conformance Database.
- Report all non-conformance situations, instrument problems, matrix problems and QC failures, which might affect the reliability of the data, to their supervisor, the Technical Director, and/or the QA Manager or member of QA staff.
- Perform 100% review of the data generated prior to entering and submitting for secondary level review.
- Suggest method improvements to their department supervisor, the Technical Director, and the QA Manager. These improvements, if approved, are incorporated. Ideas for the

optimum performance of their assigned area, for example, through the proper cleaning and maintenance of the assigned instruments and equipment, are encouraged.

- Work cohesively as a team in their department to achieve the goals of accurate results, optimum turnaround time, cost effectiveness, cleanliness, complete documentation, and personal knowledge of environmental analysis.

4.2.9 Environmental Health & Safety (EH&S) Manager

The EH&S Manager reports to the Laboratory Director and ensures that systems are maintained for the safe operation of the laboratory. For Ohio VAP, this is position is the Health & Safety Manager/Coordinator. The EH&S Manager is responsible to:

- Conduct ongoing, necessary safety training and conduct new employee safety orientation.
- Assist in developing and maintaining the Chemical Hygiene/Safety Manual.
- Administer dispersal of all Material Safety Data Sheet (MSDS) information.
- Perform regular chemical hygiene and housekeeping instruction.
- Give instruction on proper labeling and practice.
- Serve as chairman of the laboratory safety committee.
- Provide and train personnel on protective equipment.
- Oversee the inspection and maintenance of general safety equipment – fire extinguishers, safety showers, eyewash fountains, etc. and ensure prompt repairs as needed.
- Supervise and schedule fire drills and emergency evacuation drills.
- Determine what initial and subsequent exposure monitoring, if necessary to determine potential employee exposure to chemicals used in the laboratory.
- When determined necessary, conduct exposure monitoring assessments.
- Determine when a complaint of possible over-exposure is "reasonable" and should be referred for medical consultation.
- Assist in the internal and external coordination of the medical consultation/monitoring program conducted by TestAmerica's medical consultants.

4.2.10 Sample Control Manager

The Sample Control Manager reports to the Project Management Operations Manager. The responsibilities are outlined below:

- Direct the logging of incoming samples into the LIMS.
- Ensure the verification of data entry from login.

4.2.11 Project Management Operations Manager

The Project Management (PM) Operations Manager reports to the Laboratory Director and serves as the interface between the laboratory's analytical departments and the laboratory's clients. The staff consists of the Project Management team. With the overall goal of total client satisfaction, the functions of this position are outlined below:

- Technical training and growth of the PM team.
- Technical liaison for the PM team.
- Human resource management of the PM team.
- Responsible to ensure that clients receive the proper sampling supplies.
- Coordinating status meetings with departmental supervisors.
- Responsible for assistance to clients regarding the resolution of problems concerning COC.
- Ensuring that client specifications, when known, are met by communicating project and quality assurance requirements to the laboratory.
- Notifying the department supervisors of incoming projects and sample delivery schedules.
- Accountable to clients for communicating sample progress/status.
- Responsible for discussing with client any project-related problems, resolving service issues, and coordinating technical details with the laboratory staff.
- Responsible for staff familiarization with specific quotes, sample log-in review, and final report completeness.
- Monitor the status of all data package projects in-house to ensure timely and accurate delivery of reports.
- Inform clients of data package-related problems and resolve service issues.
- Coordinate requests for sample containers and other services (data packages).

4.2.12 Project Managers

The Project Management team consists of Project Managers who report to the Project Management Operations Manager. The Project Managers are responsible to:

- Monitor analytical and QA project requirements for a specified project
- Ensure that client specifications, when known, are met by communicating project and quality assurance requirements to the laboratory.
- Notify laboratory personnel of incoming projects and sample delivery schedules.
- Be accountable to clients for communicating sample progress/status.
- Ensure that clients receive the proper sampling supplies.
- Coordinate subcontract work
- Inform clients of project related problems, resolving service issues, and coordinating technical issues with the laboratory staff.
- Provide assistance to clients regarding resolution of problems concerning COC.

- Monitor the status of all projects in-house to ensure timely delivery of reports.
- Perform review of projects for completeness and compliance to client needs prior to release to client.
- Generate final laboratory reports and has signature authority (where approved).

4.2.13 Purchasing / Facilities Maintenance Manager

The Purchasing / Facilities Maintenance Manager reports to the Laboratory Director. He/She is responsible for:

- Coordinating facility maintenance, and
- Oversight of purchasing and receiving of supplies.

4.2.14 Shipping Manager / Sample Archiving and Disposal

The Shipping Manager reports to the Laboratory Director. He/She is responsible for ensuring the timely and correct shipment of sample containers, including proper preservatives and instructions, to clients. He/She maintains accurate records of sample container shipments. In addition, he/she:

- Supervises the organized storage and appropriate climate control of samples.
- Supervises the disposal of samples in accordance with the Waste Disposal SOP, the Hazardous Waste Contingency Plan in the Chemical Hygiene/Safety Manual, and the U. S. Department of Agriculture requirements.

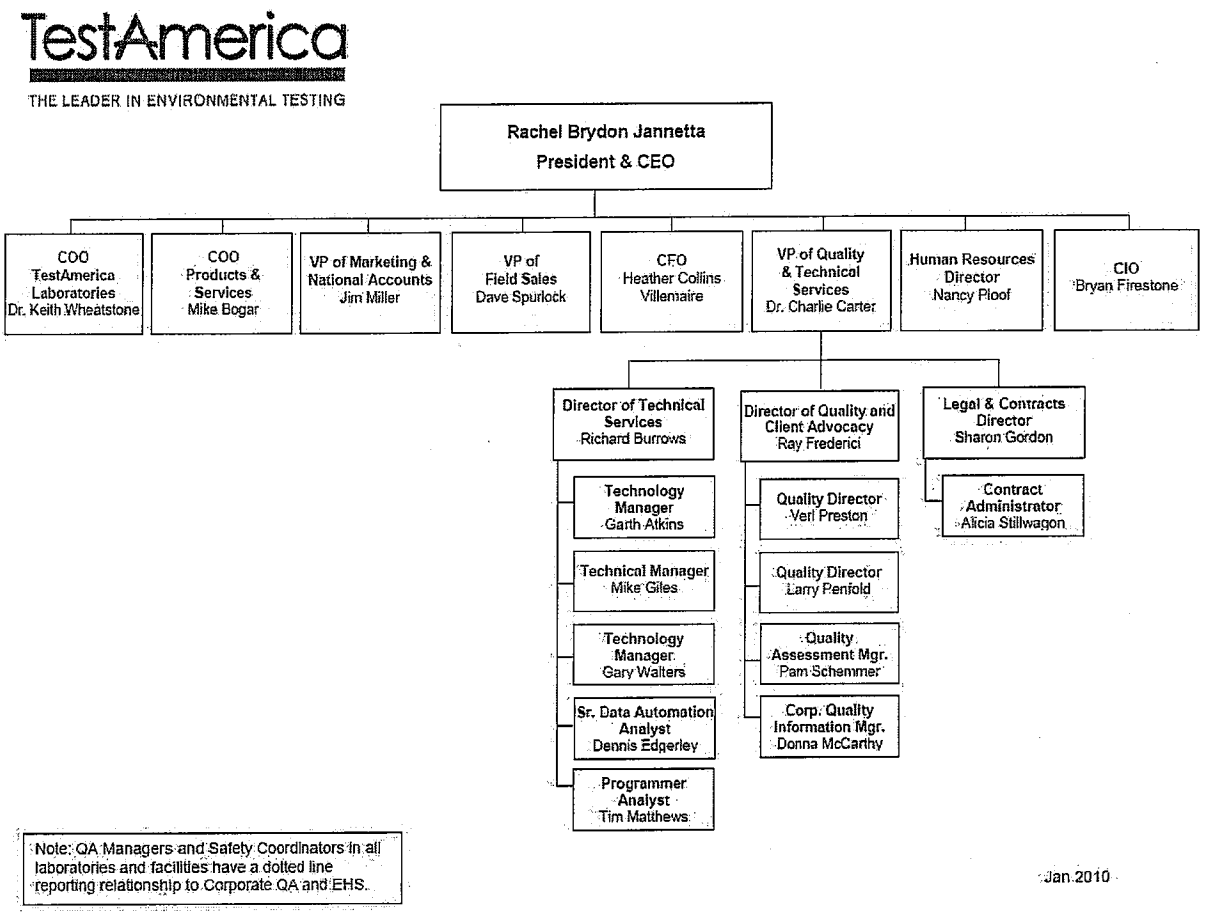
4.3 DEPUTIES

The following table defines who assumes the responsibilities of key personnel in their absence:

Key Personnel	Primary Deputy	Support Deputy
Laboratory Director	Technical Director	QA Manager PM Operations Manager
QA Manager	Laboratory Director	Technical Director
Technical Director	Laboratory Director	QA Manager Operations Managers
EHS Manager	Human Resources	Laboratory Director

Figure 4-1.

Corporate Organization Chart



SECTION 5

QUALITY SYSTEM
(NELAC 5.4.2)

5.1 QUALITY POLICY STATEMENT

It is TestAmerica's Policy to:

- Provide data of known quality to its clients by adhering to approved methodologies, ISO/IEC 17025:2005, regulatory requirements and the QA/QC protocols.
- Effectively manage all aspects of the laboratory and business operations by the highest ethical standards.
- Continually improve systems and provide support to quality improvement efforts in laboratory, administrative and managerial activities. TestAmerica recognizes that the implementation of a quality assurance program requires management's commitment and support as well as the involvement of the entire staff.
- Provide clients with the highest level of professionalism and the best service practices in the industry.
- Comply with the ISO/IEC 17025:2005 International Standard and continually improve the effectiveness of the management system.

Every staff member at TestAmerica Nashville plays an integral part in quality assurance and is held responsible and accountable for the quality of their work. It is, therefore, required that all laboratory personnel are trained and agree to comply with applicable procedures and requirements established by this document.

5.2 ETHICS AND DATA INTEGRITY

TestAmerica is committed to ensuring the integrity of its data and meeting the quality needs of its clients. The elements of TestAmerica's Ethics and Data Integrity Program include:

- An Ethics Policy (Corporate Policy No. CA-L-P-001) and Employee Ethics Statements.
- Ethics and Compliance Officers (ECOs).
- A training program.
- Self-governance through disciplinary action for violations.
- A confidential mechanism for anonymously reporting alleged misconduct and a means for conducting internal investigations of all alleged misconduct. (Corporate SOP No. CA-L-S-001)
- Procedures and guidance for recalling data if necessary (Corporate SOP No. CA-L-S-001).
- An effective external and internal monitoring system that includes procedures for internal audits (Section 16).

- Produce results, which are accurate and include QA/QC information that meets client pre-defined Data Quality Objectives (DQOs).
- Present services in a confidential, honest and forthright manner.
- Provide employees with guidelines and an understanding of the ethical and quality standards of our industry.
- Operate our facilities in a manner that protects the environment and the health and safety of employees and the public.
- Obey all pertinent federal, state and local laws and regulations and encourage other members of our industry to do the same.
- Educate clients as to the extent and kinds of services available.
- Assert competency only for work for which adequate personnel and equipment are available and for which adequate preparation has been made.
- Promote the status of environmental laboratories, their employees, and the value of services rendered by them.

5.3 QUALITY SYSTEM SUPPORTING DOCUMENTATION

The laboratory's Quality System is communicated through a variety of documents:

- Quality Assurance Manual – Each laboratory has a lab specific quality assurance manual.
- Corporate SOPs and Policies - Corporate SOPs and Policies are developed for use by all relevant laboratories. They are incorporated into the laboratory's, normal, SOP distribution, training and tracking system. Corporate SOPs may be general or technical.
- Work Instructions - A subset of procedural steps, tasks or forms associated with an operation of a management system (e. g., checklists, preformatted bench sheets, forms).
- Laboratory SOPs – General and Technical
- Corporate Quality Policy Memorandums
- Laboratory QA/QC Policy Memorandums

5.3.1 Order of Precedence

In the event of a conflict or discrepancy between policies, the order of precedence is as follows:

- Corporate Quality Policy Memorandum
- Corporate Quality Management Plan (CQMP)
- Corporate SOPs and Policies
- Laboratory QA/QC Policy Memorandum
- Laboratory Quality Assurance Manual (QAM)
- Laboratory SOPs and Policies
- Other (Work Instructions (WI), memos, flow charts, etc.)

Note: The laboratory has the responsibility and authority to operate in compliance with regulatory requirements of the jurisdiction in which the work is performed. Where the CQMP conficts with those regulatory requirements, the regulatory requirements of the jurisdiction shall hold primacy. The laboratory's QAM shall take precedence over the CQMP in those cases.

5.4 QA/QC OBJECTIVES FOR THE MEASUREMENT OF DATA

Quality Assurance (QA) and Quality Control (QC) are activities undertaken to achieve the goal of producing data that accurately characterize the sites or materials that have been sampled. Quality Assurance is generally understood to be more comprehensive than Quality Control. Quality Assurance can be defined as the integrated system of activities that ensures that a product or service meets defined standards.

Quality Control is generally understood to be limited to the analyses of samples and to be synonymous with the term *"analytical quality control"*. QC refers to the routine application of statistically based procedures to evaluate and control the accuracy of results from analytical measurements. The QC program includes procedures for estimating and controlling precision and bias and for determining reporting limits.

Request for Proposals (RFPs) and Quality Assurance Project Plans (QAPP) provide a mechanism for the client and the laboratory to discuss the data quality objectives in order to ensure that analytical services closely correspond to client needs. The client is responsible for developing the QAPP. In order to ensure the ability of the laboratory to meet the Data Quality Objectives (DQOs) specified in the QAPP, clients are advised to allow time for the laboratory to review the QAPP before being finalized. Additionally, the laboratory provides support to the client for developing the sections of the QAPP that concern laboratory activities.

Historically, laboratories have described their QC objectives in terms of precision, accuracy, representativeness, comparability, completeness, selectivity and sensitivity (PARCCSS).

5.4.1 Precision

The laboratory objective for precision is to meet the performance for precision demonstrated for the methods on similar samples and to meet data quality objectives of the EPA and/or other regulatory programs. Precision is defined as the degree of reproducibility of measurements under a given set of analytical conditions (exclusive of field sampling variability). Precision is documented on the basis of replicate analysis, usually duplicate or matrix spike (MS) duplicate samples. The calculation of precision is described in Section 25.

5.4.2 Accuracy

The laboratory objective for accuracy is to meet the performance for accuracy demonstrated for the methods on similar samples and to meet data quality objectives of the EPA and/or other regulatory programs. Accuracy is defined as the degree of bias in a measurement system. Accuracy may be documented through the use of laboratory control samples (LCS) and/or MS. A statement of accuracy is expressed as an interval of acceptance recovery about the mean recovery. The calculation of accuracy is described in Section 25.

5.4.3 Representativeness

The laboratory objective for representativeness is to provide data which is representative of the sampled medium. Representativeness is defined as the degree to which data represent a characteristic of a population or set of samples and is a measurement of both analytical and field sampling precision. The representativeness of the analytical data is a function of the procedures used in procuring and processing the samples. The representativeness can be documented by the relative percent difference between separately procured, but otherwise identical samples or sample aliquots.

The representativeness of the data from the sampling sites depends on both the sampling procedures and the analytical procedures. The laboratory may provide guidance to the client regarding proper sampling and handling methods in order to assure the integrity of the samples.

5.4.4 Comparability

The comparability objective is to provide analytical data for which the accuracy, precision, representativeness and reporting limit statistics are similar to these quality indicators generated by other laboratories for similar samples, and data generated by the laboratory over time.

The comparability objective is documented by inter-laboratory studies carried out by regulatory agencies or carried out for specific projects or contracts, by comparison of periodically generated statements of accuracy, precision and reporting limits with those of other laboratories.

5.4.5 Completeness

The completeness objective for data is 90% (or as specified by a particular project), expressed as the ratio of the valid data to the total data over the course of the project. Data is considered valid if it is adequate for its intended use. Data usability is defined in a QAPP, project scope or regulatory requirement. Data validation is the process for reviewing data to determine its usability and completeness. If the completeness objective is not met, actions must be taken internally and with the data user to improve performance. This may take the form of an audit to evaluate the methodology and procedures as possible sources for the difficulty or may result in a recommendation to use a different method.

5.4.6 Selectivity

Selectivity is defined as: The capability of a test method or instrument to respond to a target substance or constituent in the presence of non-target substances. Target analytes are separated from non-target constituents and subsequently identified/detected through one or more of the following, depending on the analytical method: extractions (separation), digestions (separation), interelement corrections (separation), use of matrix modifiers (separation), specific retention times (separation and identification), confirmations with different columns or detectors (separation and identification), specific wavelengths (identification), specific mass spectra (identification), specific electrodes (separation and identification), etc..

5.4.7 Sensitivity

Sensitivity refers to the amount of analyte necessary to produce a detector response that can be reliably detected (Method Detection Limit) or quantified (Reporting Limit).

5.5 CRITERIA FOR QUALITY INDICATORS

The laboratory prepares a Control Limits Manual containing tables that summarize the precision and accuracy acceptability limits for analyses performed at TestAmerica Nashville. The Control Limits Manual is considered to be part of the QA Manual and therefore carries the same revision number as the QA Manual. Letter suffixes are added to the revision number if it becomes necessary to update the Control Limits Manual during the course of the year prior to a QA Manual update. This summary includes an effective date, is updated each time new limits are generated and is distributed in the same manner as the QA Manual to the analytical departments. It also is available on the laboratory server. Unless otherwise noted, limits within these tables are laboratory generated. Some acceptability limits are derived from US EPA methods when they are required. Where US EPA method limits are not required, TestAmerica Nashville has developed limits from evaluation of data from similar matrices. Criteria for development of control limits are contained in Section 25.

5.6 STATISTICAL QUALITY CONTROL

Statistically-derived precision and accuracy limits are required by selected methods (such as SW-846) and programs. TestAmerica Nashville routinely utilizes statistically-derived limits to evaluate method performance and determine when corrective action is appropriate. The analysts are instructed to use the current limits in the laboratory (dated and approved by the Technical Director and QA Manager) and entered into the Laboratory Information Management System (LIMS). The Quality Assurance department maintains an archive of all limits used within the laboratory. (See SOP Archiving / SA08-194.) If a method defines the QC limits, the method limits are used.

If a method requires the generation of historical limits, the lab develops such limits from recent data in the QC database of the LIMS following the guidelines described in Section 25. All calculations and limits are documented and dated when approved and effective. On occasion, a client requests contract-specified limits for a specific project.

Surrogate recoveries are determined for a specific time period as defined above. The resulting ranges are entered in LIMS.

Current QC limits are entered and maintained in the LIMS analyte database. As sample results and the related QC are entered into LIMS, the sample QC values are compared with the limits in LIMS to determine if they are within the acceptable range. The analyst then evaluates if the sample needs to be rerun or re-extracted/rerun or if a comment is to be added to the report explaining the reason for the QC outlier.

5.6.1 QC Charts

As the QC limits are calculated, QC charts are generated showing warning and control limits for the purpose of evaluating trends. The QA Manager evaluates these to determine if adjustments need to be made or for corrective actions to methods. All findings are documented and kept on file.

5.7 QUALITY SYSTEM METRICS

In addition to the QC parameters discussed above, the entire Quality System is evaluated on a monthly basis through the use of specific metrics (refer to Section 17). These metrics are used to drive continuous improvement in the laboratory's Quality System.

SECTION 6

DOCUMENT CONTROL
(NELAC 5.4.3)

6.1 OVERVIEW

The QA Department is responsible for the control of documents used in the laboratory to ensure that approved, up-to-date documents are in circulation and out-of-date (obsolete) documents are archived or destroyed. The following documents, at a minimum, must be controlled in the lab:

- Laboratory Quality Assurance Manual
- Laboratory Standard Operating Procedures (SOP)
- Laboratory Policies
- Work Instructions and Forms
- Corporate Policies and Procedures distributed outside the intranet

Corporate Quality posts Corporate Manuals, SOPs, Policies, Work Instructions, White Papers and Training Materials on the company intranet site. These Corporate documents are only considered controlled when they are read on the company intranet site. Printed copies are considered uncontrolled unless the laboratory physically distributes them as controlled documents. A detailed description of the procedures for issuing, authorizing, controlling, distributing, and archiving Corporate documents is found in Corporate SOP No. CW-Q-S-001, Corporate Document Control and Archiving. The laboratory's internal document control procedure is defined in the following lab SOPs: Document Control of Internal Technical Forms, Work Instructions, Information Sheets / NV08-152, Archiving of Technical and Quality Department Paper Records / NV 08-194, and Organization of Quality Department Documents / NV08-220.

The laboratory QA Department also maintains access to various references and document sources integral to the operation of the laboratory. This includes reference methods and regulations. Instrument manuals (hard or electronic copies) are also maintained by the laboratory.

The laboratory maintains control of records for raw analytical data and supporting records such as audit reports and responses, logbooks, standard logs, training files, MDL studies, Proficiency Testing (PT) studies, certifications and related correspondence, and corrective action reports. Raw analytical data consists of bound logbooks, instrument printouts, any other notes, magnetic media, electronic data and final reports. Discussion on records control is described in Section 15.

The maintenance of purchasing data is discussed in Section 9.

The maintenance of sales and marketing contracts is discussed in Section 7.

6.2 DOCUMENT APPROVAL AND ISSUE

The pertinent elements of a control system for each document include a unique document name and number, the number of pages of the item, the effective date, revision number and the laboratory's name. The QA department is responsible for the maintenance of the system and maintains the items in the QA Office.

Controlled documents are authorized by the QA Department and other management. In order to develop a new document, a manager submits a paper or electronic draft to the QA Department for suggestions and approval before use. Upon approval, QA personnel add the identifying version information to the document and retain the official document on file. The official document is provided as needed to those using it. Controlled documents are available at all locations where the operational activity described in the document is performed (this may include electronic access). Controlled documents are identified as such and records of their distribution are kept by the QA Department. Document control may be achieved by either electronic or hardcopy distribution.

Handwritten edits to controlled documents are not allowed, except by QA staff. Necessary changes to controlled documents must be brought to the attention of the QA department by using the appropriate notification and amendment procedures for controlled documents.

The QA Department maintains a list of the official versions of controlled documents.

Quality System Policies and Procedures are reviewed at a minimum of every two years except for drinking water SOPs which are reviewed every year and revised as appropriate. Changes to documents occur when a procedural change warrants a revision of the document.

6.3 PROCEDURES FOR DOCUMENT CONTROL POLICY

For changes to the QA Manual, refer to SOP Document Control / NV08-152. Uncontrolled copies must not be used within the laboratory. Previous revisions and back-up data are stored by the QA department. Electronic copies are stored on the QA server.

For changes to SOPs, refer to SOP No. CW-Q-S-002, Writing a Standard Operating Procedure (SOP) and SOP Document Control / NV08-152. The QA Department has a complete file of all current and previous versions, showing changes, of each SOP. Additionally, there are controlled notebooks of current SOPs in the lab. These are updated by the QA department. There is a table of contents. Electronic versions of current, previous, and in-transition SOPs are maintained on a QA hard drive that is backed up weekly. Current, unchangeable versions are stored on the QA server/SOPs.

Changes to facilities, the QA Manual, certifications, personnel, safety/health, capabilities are documented in the Management of Change log as prescribed in Section 14.

Forms, worksheets, work instructions and information are organized by department in the QA office. There is a table of contents. Electronic versions are kept on a hard drive in the QA department; hard copies are kept in QA files. The procedure for the care of these documents is in SOP Document Control / NV08-152.

Reference books, regulations, and other external protocols are listed, with location, in the QA office. This list is updated as needed.

Logbooks and preparation worksheets are initialized and stored in an archiving system described in SOP NV08-194 for easy tracking and retrieval.

Certification correspondence, audit reports and responses, control charts, MDLs, training files, subcontractor credentials, and PT studies are stored by date in the QA office in appropriate files. These documents are not uniquely identified.

6.4 OBSOLETE DOCUMENTS

All invalid or obsolete documents are removed, or otherwise prevented from unintended use. The laboratory has specific procedures as described above to accomplish this. In general, obsolete documents are collected from employees according to distribution lists and are marked obsolete on the cover or destroyed. At least one copy of the obsolete document is archived as described in Section 15.

SECTION 7

REVIEW OF WORK REQUEST

7.1 OVERVIEW

TestAmerica Nashville has established procedures for the review of work requests and contracts, oral or written. The procedures include evaluation of the laboratory's capability and resources to meet the contract's requirements within the requested time period. All requirements, including the methods to be used, must be adequately defined, documented and understood. For many environmental sampling and analysis programs, testing design is site or program specific and does not necessarily "fit" into a standard laboratory service or product. It is TestAmerica's intent to provide both standard and customized environmental laboratory services to our clients.

A thorough review of technical and QC requirements contained in contracts is performed to ensure project success. The appropriateness of requested methods, and the lab's capability to perform them must be established. Projects, proposals and contracts are reviewed for adequately defined requirements and TestAmerica's capability to meet those requirements. Alternate test methods that are capable of meeting the clients' requirements may be proposed by the lab. A review of the lab's capability to analyze non-routine analytes is also part of this review process.

All projects, proposals and contracts are reviewed for the client's requirements in terms of compound lists, test methodology requested, sensitivity (method detection and reporting limits), accuracy, and precision requirements (% Recovery and RPD). The reviewer ensures that the laboratory's test methods are suitable to achieve these regulatory and client requirements and that the laboratory holds the appropriate certifications and approvals to perform the work. The laboratory and any potential subcontract laboratories must be certified, as required, for all proposed tests.

The laboratory must determine if it has the necessary physical, personnel and information resources to meet the contract, and if the personnel have the expertise needed to perform the testing requested. Each proposal is checked for its impact on the capacity of the laboratory's equipment and personnel. As part of the review, the proposed turnaround time will be checked for feasibility.

Electronic or hard copy deliverable requirements are evaluated against the lab's capacity for production of the documentation.

If the laboratory cannot provide all services but intends to subcontract such services, whether to another TestAmerica facility or to an outside firm, this must be documented and discussed with the client prior to contract approval. (Refer to Section 8 for Subcontracting Procedures.)

The laboratory informs the client of the results of the review if it indicates any potential conflict, deficiency, lack of accreditation, or inability of the lab to complete the work satisfactorily. Any discrepancy between the client's requirements and TestAmerica's capability to meet those requirements is resolved in writing before acceptance of the contract. It is necessary that the

contract be acceptable to both the laboratory and the client. Amendments initiated by the client and/or TestAmerica, are documented in writing.

All contracts, QAPPs, Sampling and Analysis Plans (SAPs), contract amendments, and documented communications become part of the project record.

The same contract review process used for the initial review is repeated when there are amendments to the original contract by the client, and the participating personnel are informed of the changes.

7.2 REVIEW SEQUENCE AND KEY PERSONNEL

Appropriate personnel must review the work request at each stage of evaluation.

For routine projects and other simple tasks, a review by the Project Manager (PM) is considered adequate. The PM confirms that the laboratory has any required certifications, that it can meet the clients' data quality and reporting requirements, and that the lab has the capacity to meet the clients turn around needs. Project Managers contact the appropriate laboratory management or corporate personnel if any of the details are unknown, differ from what standard payment policy and insurance coverage includes, or if the task appears to fall outside the Project Manager's job responsibilities.

For complex or large projects, the proposed contract is given to the Director of National Accounts, who decides which lab will receive the work based on the scope of work and other requirements, including certification, testing methodology, and available capacity to perform the work. The contract review process is outlined in SOP No. CA-L-P-002, Contract Compliance Policy.

This review encompasses all facets of the operation. The scope of work is distributed to the appropriate personnel, as needed based on scope of contract, to evaluate all of the requirements shown above (not necessarily in the order below):

- Legal & Contracts Director
- General Manager
- The Laboratory Project Management Operations Manager
- Laboratory and/or Corporate Technical Directors
- Regional and/or national account representatives
- Laboratory and/or Corporate Quality
- Laboratory and/or Corporate Environmental Health and Safety Managers/Directors
- The Laboratory Director reviews the formal laboratory quote and makes final acceptance for their facility.

The Director of National Accounts, Legal & Contracts Director, or local account representative then submits the final proposal to the client.

In the event that one of the above personnel is not available to review the contract, his or her back-up fulfills the review requirements.

The Legal & Contracts Director maintains copies of all signed contracts.

7.3 DOCUMENTATION

Appropriate records are maintained for every contract or work request. All stages of the contract review process are documented and include records of any significant changes.

The contract is distributed to and maintained by the appropriate sales/marketing personnel. A copy of the contract and formal quote is filed with the laboratory PM and the Lab Director.

Records are maintained of pertinent discussions with a client relating to the client's requirements or the results of the work during the period of execution of the contract. The PM keeps correspondence records (i. e., e-mail) and a phone log of conversations with the client. These records are archived by the laboratory facility for a minimum of five years.

7.3.1 Project-Specific Quality Planning

Communication of contract specific technical and QC criteria is an essential activity in ensuring the success of site specific testing programs. To achieve this goal, TestAmerica Nashville assigns a PM to each client. The PM is the first point of contact for the client. It is the PM's responsibility to ensure that project specific technical and QC requirements are effectively evaluated and communicated to the laboratory personnel before and during the project. QA department involvement may be needed to assist in the evaluation of custom requirements.

PMs are the primary client contact and they ensure resources are available to meet project requirements. Although PMs do not have direct reports or staff in production, they coordinate opportunities and work with laboratory management and supervisory staff to ensure available resources are sufficient to perform work for the client's project. Project management is positioned between the client and laboratory resources.

Prior to work on a new project, the dissemination of project information and/or project opening meetings may occur to discuss schedules and unique aspects of the project. Items to be discussed may include the project technical profile, turnaround times, holding times, methods, analyte lists, reporting limits, deliverables, sample hazards, or other special requirements. The PM introduces new projects to the laboratory staff through project kick-off meetings or to the supervisory staff during production meetings. These meetings provide direction to the laboratory staff in order to maximize production and client satisfaction, while maintaining quality. In addition, project notes may be associated with each sample batch as a reminder upon sample receipt and analytical processing.

During the project, any change that may occur within an active project is agreed upon between the client/regulatory agency and the PM/laboratory. These changes (e. g., use of a non-standard method or modification of a method) and approvals must be documented prior to implementation. Documentation pertains to any document, e. g., letter, e-mail, variance, contract addendum, which has been signed by both parties.

Such changes are also communicated to the laboratory during production meetings. Such changes are updated to the project notes and are introduced to the managers/supervisors at these

meetings. The laboratory staff is then introduced to the modified requirements via the individual laboratory operations managers/department supervisors. After the modification is implemented into the laboratory process, documentation of the modification is made in the case narrative of the data report(s).

TestAmerica strongly encourages client visits to the laboratory and formal/informal information sharing sessions with employees in order to effectively communicate ongoing client needs as well as project specific details for customized testing programs.

SECTION 8

SUBCONTRACTING OF TESTS
(NELAC 5.4.5)

8.1 OVERVIEW

For the purpose of this quality manual, the phrase subcontract laboratory refers to a laboratory external to the TestAmerica laboratory network. The phrase "work sharing" refers to internal transfers of samples between TestAmerica laboratories. The term outsourcing refers to the act of subcontracting tests.

When contracting with our clients, the laboratory makes commitments regarding the services to be performed and the data quality for the results to be generated. When the need arises to outsource testing for our clients because project scope, changes in laboratory capabilities, capacity or unforeseen circumstances, we must be assured that the subcontractors or work sharing laboratories understand the requirements and will meet the same commitments we have made to the client. Refer to the SOP on Subcontracting Procedures (CA-L-S-002) and the Work Sharing Process SOP (CA-C-S-001).

When outsourcing analytical services, the laboratory must assure, to the extent necessary, that the subcontract or work sharing laboratory maintains a program consistent with the requirements of this document, the requirements specified in NELAC/ISO 17025 and/or the client's Quality Assurance Project Plan (QAPP). All QC guidelines specific to the client's analytical program are transmitted to the subcontractor and agreed upon before sending the samples to the subcontract facility. Additionally, work requiring accreditation is placed with an appropriately accredited laboratory. The laboratory performing the subcontracted work is identified in the final report, as is non-NELAC accredited work where required.

Project Managers (PMs), Customer Service Managers (CSM), or Regional Account Executives (RAE) for the Export Lab are responsible for obtaining client approval prior to outsourcing any samples. The laboratory advises the client of a subcontract or work sharing arrangement in writing, and approval from the client (Figure 8-1) is retained in the project folder.

Note: In addition to the client, some regulating agencies, such as the US Army Corps of Engineers and the USDA, require notification prior to placing such work.

8.2 QUALIFYING AND MONITORING SUBCONTRACTORS

Whenever a PM (or Regional Account Executive (RAE) or Customer Service Manager (CSM)) becomes aware of a client requirement or laboratory need where samples must be outsourced to another laboratory, the other laboratory(s) are selected based on the following:

- The first priority is to attempt to place the work in a qualified TestAmerica laboratory;
- Firms specified by the client for the task (Documentation that a subcontractor was designated by the client must be maintained with the project file. This documentation can be as simple as placing a copy of an e-mail from the client in the project folder);
- Firms listed as pre-qualified and currently under a subcontract with TestAmerica. A listing of all approved subcontracting laboratories and supporting documentation is available on the

TestAmerica intranet site. Verify necessary accreditation for the requested tests prior to sending samples.

- Firms identified in accordance with the company's Small Business Subcontracting program as small, women-owned, veteran-owned and/or minority-owned businesses;
- NELAC or A2LA accredited laboratories.
- In addition, the firm must hold the appropriate certification to perform the work required.

All TestAmerica laboratories are pre-qualified for work sharing provided they hold the appropriate accreditations, can adhere to the project/program requirements, and the client has approved sending the samples to that laboratory. The client must provide written acknowledgement that the samples can be sent to that facility. This written acknowledgement is to be provided using the form in Figure 8-1. The originating laboratory is responsible for communicating all technical, quality, and deliverable requirements as well as other contract needs. Refer to SOP No. CA-C-S-001, Work Sharing Process.

When the potential sub-contract laboratory has not been previously approved, Account Executives or PMs may nominate a laboratory as a subcontractor based on need. The decision to nominate a laboratory must be approved by the Laboratory Director. The Laboratory Director requests that the QA Manager begin the process of approving the subcontract laboratory as outlined in Corporate SOP No. CA-L-S-002, Subcontracting Procedures. The client must provide written acknowledgement that the samples can be sent to that facility. This written acknowledgement is to be provided using the form in Figure 8-1.

8.2.1 Once the appropriate accreditation and legal information is received by the laboratory, it is evaluated for acceptability (where applicable) by lab management and forwarded to Corporate Contracts for formal contracting with the laboratory. Corporate Contracts adds the lab to the approved subcontractor list on the intranet site along with the associate documentation and notifies the finance group for JD Edwards.

8.2.2 The client assumes responsibility for the quality of the data generated from the use of a subcontractor they have requested the lab to use. The qualified subcontractors on the intranet site are known to meet minimal standards. TestAmerica does not certify laboratories. The subcontractor is on our approved list and can only be recommended to the extent that we would use them.

8.2.3 The status and performance of qualified subcontractors are monitored periodically by the Corporate Contracts and/or Quality Department. Any problems identified are brought to the attention of TestAmerica's Corporate Finance or Corporate Quality personnel.

- Complaints must be investigated. Documentation of the complaint, investigation and corrective action is maintained in the subcontractor's file on the intranet site. Complaints are posted using the Vendor Performance Report (Form No. CW-F-WI-009).
- Information is updated on the intranet when new information is received from the subcontracted laboratories.
- Subcontractors in good standing are retained on the intranet listing. The lab QA Manager notifies all TestAmerica laboratories and Corporate Quality and Corporate Contracts if any laboratory requires removal from the intranet site. This notification is posted on the intranet

site and e-mailed to all Lab Directors/Managers, QA Managers and Sales Personnel.

8.3 OVERSIGHT AND REPORTING

The PM must request that the selected subcontractor be presented with a subcontract, if one is not already executed between the laboratory and the subcontractor. The subcontract must include terms which reflect the requirements of our clients, either in the subcontract itself or through the mechanism of work orders relating to individual projects. A standard subcontract and the Lab Subcontractor Vendor Package (posted on the intranet) can be used to accomplish this, and the Legal & Contracts Director can tailor the document or assist with negotiations, if needed. The PM (or RAE or CSM, etc) responsible for the project must advise and obtain client consent to the subcontract as appropriate, and provide the scope of work to ensure that the proper requirements are made a part of the subcontract and are made known to the subcontractor.

Prior to sending samples to the subcontracted laboratory, the PM confirms their certification status to determine if it is current and scope-inclusive. The information is documented on a Subcontracted Sample Form (Figure 8-2) and the form is retained in the project folder. For TestAmerica laboratories, certifications can be viewed on the company's Total Assess Database.

The Shipping department is responsible for ensuring compliance with QA requirements and applicable shipping regulations when shipping samples to a subcontracted laboratory.

All subcontracted samples must be accompanied by a Chain of Custody (COC). A copy of the original COC sent by the client must be included with all samples subbed within TestAmerica.

Through communication with the subcontracted laboratory, the PM monitors the status of the subcontracted analyses, facilitates successful execution of the work, and ensures the timeliness and completeness of the analytical report.

Non-NELAC accredited work must be identified in the subcontractor's report as appropriate. If NELAC accreditation is not required, the report does not need to include this information.

Reports submitted from subcontractor laboratories are not altered and are included in their original form in the final project report. This clearly identifies the data as being produced by a subcontractor facility. If subcontract laboratory data is incorporated into the laboratory's EDD (i. e., imported), the report must explicitly indicate which lab produced the data for which methods and samples.

Note: The results submitted by a TestAmerica work sharing laboratory may be transferred electronically, and the results reported by the TestAmerica work sharing lab are identified on the final report. The report must explicitly indicate which lab produced the data for which methods and samples. The final report must include a copy of the completed COC for all work sharing reports.

8.4 CONTINGENCY PLANNING

The Laboratory Director may waive the full qualification of a subcontractor process temporarily to meet emergency needs. In the event this provision is utilized, the PM must verify certifications and notify the laboratory QA Manager of this event.

The comprehensive subcontractor approval process, as outlined in Corporate SOP No. CA-L-S-002, Subcontracting Procedures must then be initiated within 30 calendar days of subcontracting.

Figure 8-1.

Client-Approved Subcontractor Form

Client Information:

Client Name & Account Number:_____

Client Contact: _____

Client Address: _____

_____

Project Information: (Please choose all applicable.)

- ❖ **Certification required:** □ **State** □ **NELAC** □ **A2LA** □ **AIHA**

  □ **Method**_____□ **Target compound**_____□ **Other**_____

- ❖ **Required Turn around time (method provisional)**_____

Subcontractor's Information:

Subcontractor's Name: _____

Subcontractor's Contact: _____

Subcontractor's Email: _____

Subcontractor's Address: _____

_____

Subcontractor's Phone Number: _____

Analytical Test/Compound/Method to be subcontracted: _____

_____

□ **I, the client, have designated this subcontractor for my project(s).**

□ **TestAmerica – Nashville has designated this subcontractor for my project(s).**

Certification Statement:

I hereby give ***TestAmerica Nashville*** permission to use the above noted subcontractor for the above noted testing procedures/methods. I realize that the above subcontractor will be held liable for the validity of the above mentioned testing procedures/methods. All subcontractors must meet the requirements as spelled out in project information and will follow all analytical holding times and turn around times for analytical reports. The subcontract laboratory, and not TestAmerica, will be held liable for liquidated damages for delays in subcontracted analytical reports and/or electronic data deliverables.

_____

Client Signature **Date**

Figure 8-2.

Subcontracted Sample Form

Date/Time: _____

Subcontracted Laboratory Information:

- Subcontractor's Name: _____
- Subcontractor Point of Contact: _____
- Subcontractor's Address: _____
- Subcontractor's Phone: _____
- Analyte/Method: _____
- Certified for State of Origin: _____
- NELAC Accredited: Yes_____No_____
- A2LA (or ISO 17025) Accredited: Yes_____No_____
- AIHA Accredited: Yes_____No_____
- CLP-like Required: Yes_____No_____
  (Full doc required)
- Requested Sample Due Date: _____
  (Must be put on COC)

Project Manager: _____

Laboratory Sample # Range: _____
(Only of Subcontracted Samples)

Laboratory Project Number (Billing Control #): _____

All subcontracted samples are to be sent via bonded carrier and Priority Overnight. Please attach tracking number below and maintain these records in the project files.

PM Signature_____**Date**_____

SECTION 9

PURCHASING SERVICES AND SUPPLIES
(NELAC 5.4.6)

9.1 OVERVIEW

Evaluation and selection of suppliers and vendors are performed, in part, on the basis of the quality of their products, their ability to meet the demand for their products on a continuous and short term basis, the overall quality of their services, their past history, and competitive pricing. This is achieved through evaluation of objective evidence of quality furnished by the supplier, which can include certificates of analysis, recommendations, and proof of historical compliance with similar programs for other clients. To ensure that quality critical consumables and equipment conform to specified requirements, all purchases from specific vendors are approved by a member of the supervisory or management staff. Capital expenditures are made in accordance with TestAmerica's Corporate Controlled Purchases Procedure, CW-F-S-007.

Contracts are signed in accordance with TestAmerica's Corporate Authorization Matrix Policy, CW-F-P-002. Request for Proposals (RFP's) are issued where more information is required from the potential vendors than just price. Process details are available in TestAmerica's Corporate Procurement and Contracts Policy (Policy No. CW-F-P-004). RFP's allow TestAmerica to determine if a vendor is capable of meeting requirements such as supplying all of the TestAmerica facilities, meeting required quality standards and adhering to necessary ethical and environmental standards. The RFP process also allows potential vendors to outline any additional capabilities they may offer.

Non-capital expenditure items are purchased through the requisition and approval process in JD Edwards or through other TestAmerica authorized methods (approved web-sites, purchasing cards). Labs have the ability to select from the approved vendors in JD Edwards.

9.2 GLASSWARE

Glassware used for volumetric measurements must be Class A, or verified for accuracy by the manufacturer or according to laboratory procedure. Pyrex (or equivalent) glass is used where possible. For safety purposes, thick-wall glassware is used where available.

9.2.1 Glassware Cleaning

The proper technique for cleaning glassware depends upon the intended use of the glassware being cleaned. The goal is to remove all substances from the glassware that might interfere with the analysis. Water-soluble substances can be removed with tap water followed with multiple rinses with reagent water. In some instances, detergent may be required. Detergent washings are followed by rinses with analyte-free water. Specific guidelines can be found in Table 9-1.

9.2.2 Glassware Storage

Once cleaned, glassware is capped, inverted or covered for storage in a designated cabinet, away from bulk chemicals or reagents.

9.3 REAGENTS, STANDARDS & SUPPLIES

Purchasing guidelines for equipment and reagents must meet the requirements of the specific method and testing procedures for which they are being purchased. Solvents and acids are pre-tested in accordance with Corporate SOP on Solvent & Acid Lot Testing & Approval, SOP No. CA-Q-S-001.

9.3.1 Purchasing

Chemical reagents, solvents, glassware, and general supplies are ordered as needed to maintain sufficient quantities on hand. Materials used in the analytical process must be of a known quality. The wide variety of materials and reagents available makes it advisable to specify recommendations for the name, brand, and grade of materials to be used in any determination. This information is contained in the method SOP.

The analyst provides the item number, item description, package size, and the quantity needed to his/her department supervisor. Any substitution of items requires the approval of the Technical Director, QA Manager, or Lab Director, using a Supply Item Substitution Approval Form as described in SOP Standards Control / NV08-214. Completed Supply Item Substitution forms are filed with the Purchasing Manager. The department supervisors and the Purchasing Manager place the orders. See SOP Purchasing / NV08-232 for further details.

9.3.2 Receiving

It is the responsibility of the purchasing manager to receive the shipment. It is the responsibility of the analyst who ordered the materials to date the material when received. Once the ordered reagents or materials are received, the analyst compares the information on the label or packaging to the original order to ensure that the purchase meets the quality level specified in the SOP and meets applicable specifications described below. Material Safety Data Sheets (MSDSs) are available online through the Company's intranet website. Anyone may review these for relevant information on the safe handling and emergency precautions of on-site chemicals.

9.3.3 Specifications

All methods in use in the laboratory specify the grade of reagent that must be used in the procedure. If the quality of the reagent is not specified, it may be assumed that it is not significant in that procedure and, therefore, any grade reagent may be used. It is the responsibility of the analyst to check the procedure carefully for the suitability of grade of reagent.

Chemicals must not be used past the manufacturer's expiration date and must not be used past the expiration time noted in a method SOP or the SOP Standards Control / NV08-214. If expiration dates are not provided, the laboratory may contact the manufacturer to determine an expiration date.

The laboratory assumes a five year expiration date on inorganic dry chemicals unless noted otherwise by the manufacturer or by the reference source method. Chemicals must not be used past the manufacturer's or SOP's expiration date unless 'verified'.

- An expiration date cannot be extended if the dry chemical is discolored or appears otherwise physically degraded, the dry chemical must be discarded.

- Expiration dates can be extended if the dry chemical is found to be satisfactory based on acceptable performance of quality control samples (Continuing Calibration Verification (CCV), Blanks, Laboratory Control Sample (LCS), etc.).

- If the dry chemical is used for the preparation of standards, the expiration dates can be extended 6 months if the dry chemical is compared to an unexpired independent source in performing the method and the performance of the dry chemical is found to be satisfactory. The comparison must show that the dry chemical meets CCV limits. The comparison studies are maintained by the department supervisor.

Wherever possible, standards must be traceable to national or international standards of measurement or to national or international reference materials. Records to that effect are available to the user.

Compressed gases in use are checked for pressure and secure positioning daily. The minimum total pressure must be 500 psig or the tank must be replaced. The quality of the gases must meet method or manufacturer specification or be of a grade that does not cause any analytical interference.

Water used in the preparation of standards or reagents must have a specific conductivity of less than 10 microohm-cm at 25°C. An in-line resistivity meter check is performed and recorded daily, and the conductivity is checked and recorded weekly. If the water's conductivity is greater than the specified limit, the Technical Director must be notified immediately in order to notify all departments, decide on cessation (based on intended use) of activities, and make arrangements for correction.

The laboratory may purchase reagent grade (or other similar quality) water for use in the laboratory. This water must be certified "clean" by the supplier for all target analytes or otherwise verified by the laboratory prior to use. This verification is documented.

Standard lots are verified before first time use if the laboratory switches manufacturers or has historically had a problem with the type of standard.

Purchased VOA vials must be certified clean, and the certificates must be maintained. If uncertified VOA vials are purchased, all lots must be verified clean prior to use. This verification must be maintained.

Records of manufacturer's certification and traceability statements are maintained in files or binders in each laboratory section. These records include date of receipt, lot number (when applicable) and expiration date (when applicable). Incorporation of the item into the record indicates that the analyst has compared the new certificate with a previous one for the same purpose and that no difference is noted, unless approved and so documented by the Technical Director or Quality Assurance Manager.

9.3.4 Storage

Reagent and chemical storage is important from the aspects of both integrity and safety. Light-sensitive reagents may be stored in brown-glass containers. Storage conditions are per the Corporate Environmental Health & Safety Manual (Corp. Doc. No. CW-E-M-001) and method SOPs or manufacturer instructions. Table 9-2 details general storage instructions for reagents and chemicals. Section 22 discusses conditions for standard storage.

9.4 PURCHASE OF EQUIPMENT/INSTRUMENTS/SOFTWARE

When a new piece of equipment is needed, either for additional capacity or for replacing inoperable equipment, the analyst or department supervisor makes a supply request to the Technical Director and/or the Laboratory Director. If they agree with the request, the procedures outlined in TestAmerica's Corporate Policy No. CA-T-P-001, Qualified Products List, are followed. A decision is made as to which piece of equipment can best satisfy the requirements. The appropriate written requests are completed, and purchasing places the order.

Upon receipt of a new or used piece of equipment, it is assigned a short name (such as HP-20) added to the equipment list (described in Section 21) that is maintained by the QA Department, and IT must be notified so that it can be linked to the server for back-ups. Its capability is assessed to determine if it is adequate or not for the specific application. For instruments, a calibration curve is generated, followed by MDLs, Demonstration of Capabilities (DOCs), and other relevant criteria (see Section 20). For software, its operation must be deemed reliable. Evidence of instrument verification must be retained at the instrument and in the QA Department. Software certificates supplied by the vendors are filed with the IT department. The manufacturer's operation manual is retained at the bench.

9.5 SERVICES

Service to analytical instruments (except analytical balances) is performed on an as needed basis. Routine preventive maintenance is discussed in Section 21. The need for service is determined by analysts and/or Department Managers/Supervisors. The service providers who perform the services are approved by the Department Managers/Supervisors/Technical Director.

9.6 SUPPLIERS

TestAmerica selects vendors through a competitive proposal / bid process, strategic business alliances or negotiated vendor partnerships (contracts). This process is defined in the Corporate Finance documents on Vendor Selection (SOP No. CW-F-S-018) and Procurement & Contracts Policy (Policy No. CW-F-P-004). The level of control used in the selection process is dependent on the anticipated expenses and the potential impact on TestAmerica business. Vendors that provide test and measuring equipment, solvents, standards, certified containers, instrument related service contracts or subcontract laboratory services are subject to more rigorous controls than vendors that provide off-the-shelf items of defined quality that meet the end use requirements. The JD Edwards purchasing system includes all suppliers /vendors that have been approved for use.

Evaluation of suppliers is accomplished by ensuring the supplier ships the product or material ordered and that the material is of the appropriate quality. This is documented by signing off on packing slips or other supply receipt documents. The purchasing documents contain the data that adequately describe the services and supplies ordered.

Any issues of vendor performance are to be reported immediately by the laboratory staff to the Corporate Purchasing Group by completing a Vendor Performance Report (CW-F-WI-009).

The Corporate Purchasing Group works through the appropriate channels to gather the information required to clearly identify the problem and contacts the vendor to report the problem and to make any necessary arrangements for exchange, return authorization, credit, etc.

As deemed appropriate, the Vendor Performance Reports are summarized and reviewed to determine corrective action necessary, or service improvements required by vendors

The laboratory has access to a master listing of all approved suppliers of critical consumables, supplies and services. This information is provided through the JD Edwards purchasing system.

9.6.1 New Vendor Procedure

TestAmerica employees who wish to request the addition of a new vendor must complete a J.D. Edwards Vendor Add Request Form (CW-F-WI-007 – refer to Figure 9-1), available on the intranet site. New vendors to be used for technical purchases must also undergo vendor evaluation as described in laboratory SOP Purchasing, NV08-232.

New vendors are evaluated based upon criteria appropriate to the products or services provided as well as their ability to provide those products and services at a competitive cost. Vendors are also evaluated to determine if there are ethical reasons or potential conflicts of interest with TestAmerica employees that would make it prohibitive to do business with them as well as their financial stability. The Corporate QA Department and/or the Corporate Technology Director are consulted with vendor and product selection that have an impact on quality.

Table 9-1.

Laboratory Glassware Cleaning Procedures

Analysis/Parameter	**Cleaning Procedure** (in specified order)
Extractable Organics (including Pesticides and Herbicides)	13, 1, 2, 3, 4, 5 or 7, (6 or 8 optional), 14, 15
Purgeable Organics	7, 11
Trace Metals	1, 2, 3, 4, 10, 4, 9, 3, 4
Teflon™ Microwave Vessels	3, 9, 4
Nutrients (except P) and other Wet Chemistry	1, 2, 4, 9, 4
Minerals, Demand, CN and Phenols	1, 2, 4
Residues	1, 2, 4, 12
Phosphorus	9, 1, 2 (Alconox™), 4

Key to laboratory glassware cleaning procedures:

1 Remove all labels.
2 Wash with hot tap water, a brush to scrub inside glassware and stopcocks and other small pieces, if possible, using a suitable laboratory-grade detergent:
Organics – Liquinox™, Alconox™ or equivalent,
Inorganic Anions – Liquinox™ or equivalent,
Inorganic Cations – Liquinox™, Acationox™, Micro or equivalent.
3 Rinse thoroughly with hot tap water.
4 Rinse thoroughly with deionized water.
5 Rinse thoroughly with pesticide-grade acetone.
6 Rinse thoroughly with pesticide-grade methylene chloride.
7 Rinse thoroughly with pesticide-grade methanol.
8 Rinse thoroughly with pesticide-grade hexane.
9 Rinse or soak with 1:1 HCl.
10 Rinse or soak with 10% HNO_3.
11 Bake at 105°C for 3-4 hours (Note: Class A volumetric glassware must not be baked.)
12 Bake crucibles at 105°C or 180°C for 1 hour (prior to use as per method).
13 After use, rinse with solvent used.
14 Store inverted or capped with suitable material or container stopper.
15 Rinse with solvent used in analysis as the last step prior to use.

Table 9-2.

Storage of Reagents and Chemicals

Chemical	Storage Requirements
Concentrated Acids and Bases	Stored in the original containers at room temperature. All organic acids must be stored separately from inorganic acids. Acids must not be stored with bases.
Standards for metals analysis	Stored at room temperature in the standards cabinet or a drawer near the instrument in the metals department.
Standards for extractable organics	Stored in freezers at temperatures below 0°C in the department (except DRO department standards which are stored refrigerated at <4 ± 2°C.
Standards for volatile organics	Neat standards are stored at room temperature in the standard cabinet in the department. Stock solutions and working solutions are stored in the freezer (-10 to -20°C).
Bulk Dry Chemicals	Stored at room temperature in the reagent storage area of the primary analytical department.
Working Solutions Containing Organic Compounds	Stored in the departmentas per method recommendation/ requirement. They are generally stored refrigerated at 4± 2°C.
Working Solutions Containing Only Inorganics	Stored at room temperature in the department; refrigeration is optional.
Flammable Solvents	Stored in solvent cabinets at room temperature in the organic extraction laboratory.
Non-Flammable Solvents	Stored separately from the flammable solvents in cabinets at room temperature in the organic extraction laboratory.

Figure 9-1. Example – JD Edwards Vendor Add Request Form

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JD Edwards Vendor Add Request Form

Vendor name:	Lab location and individual making request:
Vendor address (remit to):	Vendor phone:
Vendor address (remit to):	Vendor fax:
Contact name:	Product / service provided:

Reason for Vendor Addition: Check all reasons that apply

☐ Cost Reduction	Estimated Annual Savings $
☐ Replace Current Vendor	Reason?
	Vendor being Replaced?
☐ New Product / Service	Describe:
☐ ISO Approved (Required for Aerotech / P&K only)	

Small Business:

Does this vendor help us to meet our small business objectives: _____

If yes, which category: _____

Personal and Ethical Considerations:

Is there any personal conflict of interest with a TestAmerica employee and the vendor listed above? _____

Have ethical considerations been taken into account in your evaluation of this vendor?_____

Can this product be sourced from another TestAmerica facility?_____

Please complete form and email to NCPurchasing@testamericainc.com or fax to (330) 966-9275.

I approve the addition of this vendor:

_____ _____

Purchasing Manager - Patrick Eckman Corporate Controller - Leslie Bowers

Form No. CW-F-WI-007

SECTION 10

SERVICE TO THE CLIENT
(NELAC 5.4.7)

10.1 OVERVIEW

TestAmerica Nashville cooperates with clients and their representatives to monitor the laboratory's performance in relation to work performed for the client. It is the laboratory's goal to meet all client requirements in addition to statutory and regulatory requirements discussed in Section 5. The laboratory has procedures to ensure confidentiality to clients (Section 16 and 26).

Note: ISO 17025/NELAC 2003 states that a laboratory "shall afford clients or their representatives cooperation to clarify the client's request." This topic is discussed in Section 7.

10.2 SPECIAL SERVICES

The laboratory's standard procedures for reporting data are described in Section 26. Special services are also available and provided upon request. These services include:

- Reasonable access for our clients or their representatives to the relevant areas of the laboratory for the witnessing of tests performed for the client.
- Assist client-specified third party data validators as specified in the client's contract.
- Supplemental information pertaining to the analysis of their samples. Note: An additional charge may apply for additional data/information that was not requested prior to the time of sample analysis or previously agreed upon.

10.3 CLIENT COMMUNICATION

Project managers are the primary communication link to the clients. The lab informs its clients of any delays in project completion as well as any non-conformances in either sample receipt or sample analysis. Project management maintains ongoing client communication throughout the entire client project.

The Technical Director is available to discuss any technical questions or concerns that the client may have.

10.4 REPORTING

The laboratory works with the client to produce any special communication reports required by the contract.

10.5 CLIENT SURVEYS

The laboratory assesses both positive and negative client feedback. The results are used to improve overall laboratory quality and client service.

TestAmerica Nashville participates in the American Council of Independent Laboratories (ACIL) Seal of Excellence program. This program includes the submission of a survey to laboratory clients. The clients send their responses directly to ACIL.

TestAmerica's Sales and Marketing teams periodically develops lab and client specific surveys to assess client satisfaction.

SECTION 11

COMPLAINTS
(NELAC 5.4.8)

11.1 OVERVIEW

TestAmerica Nashville believes that effective client complaint handling processes have important business and strategic value. Listening to and documenting client concerns captures "client knowledge" that enables our operations to continually improve processes and client satisfaction. An effective client complaint handling process also provides assurance to the data user that the laboratory stands behind its data, service obligations and products.

A client complaint is any expression of dissatisfaction with any aspect of our business services (e.g, communications, responsiveness, data, reports, invoicing and other functions) expressed by any party, whether received verbally or in written form. Client inquiries, complaints or noted discrepancies are documented, communicated to management, and addressed promptly and thoroughly.

The laboratory has procedures for addressing both external and internal complaints, with the goal of providing satisfactory resolution to complaints in a timely and professional manner.

The nature of the complaint is identified, documented and investigated, and an appropriate action is determined and taken. In cases where a client complaint indicates that an established policy or procedure was not followed, the QA Department must evaluate whether a special audit must be conducted to assist in resolving the issue. A written confirmation or letter to the client, outlining the issue and response taken is recommended as part of the overall action taken.

The process of complaint resolution and documentation utilizes the procedures outlined in Section 13 (Corrective Actions) and is documented in the Project Information and Problem Electronic (PIPE) non-conformance database or in a Corrective Action Report (CAR).

11.2 EXTERNAL COMPLAINTS

An employee that receives a complaint initiates the complaint resolution process and the documentation of the complaint.

Complaints fall into two categories: correctable and non-correctable. An example of a correctable complaint would be one where a report re-issue would resolve the complaint. An example of a non-correctable complaint would be one where a client complains that their data was repeatedly late. Non-correctable complaints are those where the immediate issue that resulted in the complaint cannot be altered, but reoccurrences may be addressed. Non-correctable complaints are reviewed for preventive action measures to reduce the likelihood of future occurrence and mitigation of client impact.

The general steps in the complaint handling process are:

- Receiving and Documenting Complaints
- Complaint Investigation and Service Recovery

- Process Improvement

Complaints related to analytical reports are generally investigated by a Project Manager. These types of complaints may include, but are not limited to: report content and/or format, potential errors, turnaround time, bottle order shipping errors, and compliance with project requirements. The investigation may include discussions with the analyst, QA Manager, Laboratory Director, or Technical director, and is documented in a Customer Non-conformance Report (Figure 11-1) in the PIPE database.

Complaints related to quality systems, accreditation issues, and audit findings are investigated by the QA Manager. These complaints are documented in the CAR database.

The laboratory informs the initiator of the complaint of the results of the investigation and the corrective action taken, if any.

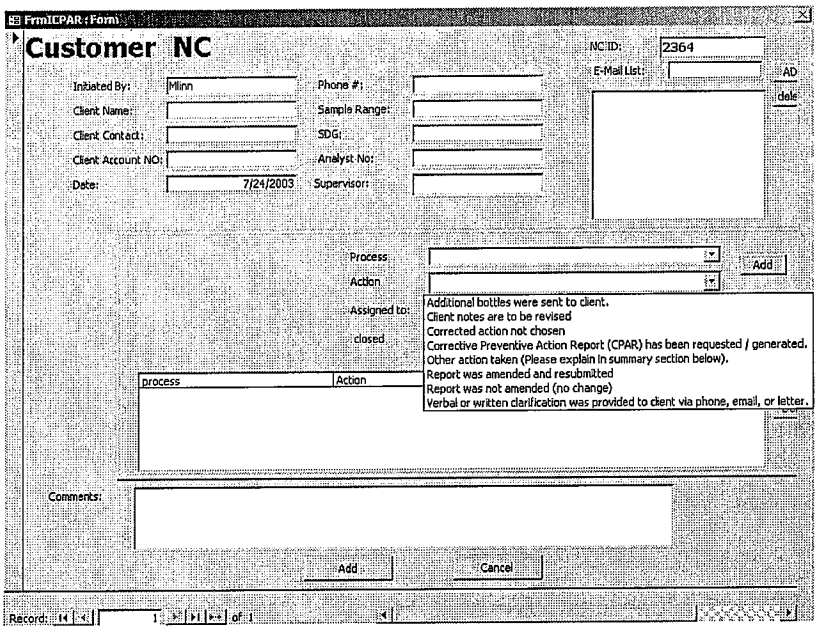
11.3 INTERNAL COMPLAINTS

Internal complaints include, but are not limited to: errors and non-conformances, training issues, internal audit findings, and deviations from methods. Corrective actions may be initiated by any staff member who observes a nonconformance. The procedures that must be followed regarding corrective action generation are outlined in Section 13. In addition, corporate management, Sales and Marketing, and Information Technology (IT) may initiate a complaint by contacting the laboratory management staff or through the corrective action system described in Section 13.

11.4 MANAGEMENT REVIEW

The number and nature of client complaints are reported by the QA Manager to the laboratory and Corporate QA Director in the QA Monthly report. Monitoring and addressing the overall level and nature of client complaints and the effectiveness of the solutions are part of the Annual Management Review (Section 17)

Figure 11-1 **Customer Non-Conformance Report**



Process Drop-down Menu
Add compounds to the report if possible
Add Facility ID Number to Analytical Report
Alter Reporting Limits
Bottle Order did not arrive or arrived late
Bottle Order received incomplete/incorrect
Change Project Name
Change Project Number
Change Sample Collection Date/Time
Change Sample ID
Change Sampler Name
Change Test Method Reported if possible
Client did not receive email version of data
Client did not receive the fax/hardcopy data
Client Notes/Special Instructions were not followed
COC requested analysis not reported
Compound missing for QC section of Analytical Report (See below)
Correct units not used in report
Detailed case narrative required
Hardcopy data vs. EDD does not agree
Incorrect pricing applied to invoice (test:_____)
Other ANALYTICAL (See Comments)
Other NON-ANALYTICAL (See Comments
Remove compounds not requested from report
Report analytical values to MDL
Report limits not achieved
Unprofessional attitude by Lab Personnel
Verify analysis (outside historical bounds)
Verify sample matrix type (solid vs. liquid)

SECTION 12

CONTROL OF NON-CONFORMING WORK
(NELAC 5.4.9)

12.1 OVERVIEW

When data discrepancies are discovered or deviations and departures from laboratory standard procedures, policies and/or client requests have occurred, corrective action is taken immediately. First, the laboratory evaluates the significance of the nonconforming work. Then, a corrective action plan is initiated based on the outcome of the evaluation. If it is determined that the nonconforming work is an isolated incident, the plan could be as simple as adding a qualifier to the final results and/or making a notation in the case narrative. If it is determined that the nonconforming work is a systematic or improper practices issue, the corrective action plan could include a more in depth investigation and a possible suspension of an analytical method. In all cases, the actions taken are documented using the laboratory's corrective action system (refer to Section 13).

Due to the frequently unique nature of environmental samples, sometimes departures from documented policies and procedures are needed. When an analyst encounters such a situation, the problem is presented to the supervisor for resolution. The supervisor may elect to discuss it with the Technical Director or have a representative contact the client to decide on a logical course of action. Once an approach is agreed upon, the analyst documents it using the laboratories corrective action system described in Section 13. This information can then be supplied to the client in the form of a footnote or a case narrative with the report. [Note: For Ohio VAP, the laboratory must not deviate from approved SOPs or the QA Manual.]

Project Management may encounter situations where a client may request that a special procedure be applied to a sample that is not standard lab practice. Based on a technical evaluation, the lab may accept or opt to reject the request based on technical or ethical merit. An example might be the need to report a compound that the lab does not normally report. The lab would not have validated the method for this compound following the procedures in Section 20. The client may request that the compound be reported based only on the calibration. Such a request would need to be approved by the Technical Director and QA Manager, documented and included in the project folder. Deviations **must** also be noted on the final report with a statement that the compound is not reported in compliance with NELAC (or the analytical method) requirements and the reason. Data being reported to a non-NELAC state would need to note the change made to how the method is normally run.

On a monthly basis, the laboratory management team reviews the non-conformance corrective actions to determine if any trends are present. If trends are found, such as repeated occurrences, further corrective action is taken to eliminate the reoccurrences as outline in Section 13.

12.2 RESPONSIBILITIES AND AUTHORITIES

TestAmerica's Corporate SOP entitled *Internal Investigation of Potential Data Discrepancies and Determination for Data Recall* (SOP No. CA-L-S-001), outlines the general procedures for the reporting and investigation of data discrepancies and alleged incidents of misconduct or

violations of TestAmerica's data integrity policies as well as the policies and procedures related to the determination of the potential need to recall data.

Under certain circumstances the Laboratory Director, the Technical Director, a Department Supervisor, or the QA Manager may authorize departures from documented procedures or policies. The departures may be a result of procedural changes due to the nature of the sample; a one-time procedure for a client; QC failures with insufficient sample to reanalyze, etc. In most cases, the client must be informed of the departure prior to the reporting of the data. Any departures must be well documented using the laboratory's corrective action procedures described in Section 13. This information may also need to be documented in logbooks and/or data review checklists and in LIMS, as appropriate. Any impacted data must be referenced in a case narrative and/or flagged with an appropriate data qualifier.

Any misrepresentation or possible misrepresentation of analytical data discovered by any laboratory staff member must be reported to facility senior management within 24 hours. The Senior Management staff is comprised of the Laboratory Director, the QA Manager, and the Department Supervisors. The reporting of issues involving alleged violations of the company's Data Integrity or Manual Integration procedures must be conveyed to an Ethics and Compliance Officer (ECO), the Director of Quality & Client Advocacy, and Corporate Quality Director within 24 hours of discovery.

Whether an inaccurate result was reported due to calculation or quantitation errors, data entry errors, improper practices, or failure to follow SOPs, the data must be evaluated to determine the possible effect.

The Laboratory Director, QA Manager, Ethics and Compliance Officers, Corporate Quality, Chief Operating Officer, General Managers and the Corporate Quality Directors have the authority and responsibility to halt work, withhold final reports, or suspend an analysis for due cause as well as authorize the resumption of work.

12.3 EVALUATION OF SIGNIFICANCE AND ACTIONS TAKEN

For each nonconforming issue reported, an evaluation of its significance and the level of management involvement needed is made. This includes reviewing its impact on the final data, whether or not it is an isolated or systematic issue, and how it relates to any special client requirements.

TestAmerica's Corporate Data Investigation & Recall Procedure (SOP No. CA-L-S-001) distinguishes between situations when it would be appropriate for the laboratory QA Manager and Laboratory Director (or his/her designee) to make the decision on the need for client notification (written or verbal) and data recall (report revision) and when the decision must be made with the assistance of the ECOs and Corporate Management. Laboratory level decisions are documented and approved using the laboratory's standard nonconformance/corrective action reporting (Section 13) in lieu of the data recall determination form contained in TestAmerica's Corporate SOP No. CA-L-S-001.

12.4 PREVENTION OF NONCONFORMING WORK

If it is determined that the nonconforming work could recur, further corrective actions must be made following the laboratory's corrective action system (Section 13).

On a monthly basis, the Senior Management team evaluates non-conformances to determine if any nonconforming work has been repeated multiple times. If so, the laboratory's corrective action process is followed.

12.5 METHOD SUSPENSION/RESTRICTION (STOP WORK PROCEDURES)

In some cases it may be necessary to suspend/restrict the use of a method or target compound which constitutes significant risk and/or liability to the laboratory. Suspension/restriction procedures can be initiated by any of the persons noted in Section 12.2, Paragraph 5 above.

Prior to suspension/restriction, confidentiality must be respected, and the problem and the required corrective and preventive action must be stated in writing and presented to the Laboratory Director.

The Laboratory Director must arrange for the appropriate personnel to meet with the QA Manager as needed. This meeting must be held to confirm that there is a problem, that suspension/restriction of the method is required and must be concluded with a discussion of the steps necessary to bring the method/target or test fully back on line. In some cases a formal group meeting may not be necessary if all appropriate personnel have already agreed there is a problem and there is agreement on the steps needed to bring the method, target or test fully back on line.

The QA Manager also initiates a corrective action report as described in Section 13 if one has not already been started. A copy of any meeting notes and agreed upon steps are faxed or e-mailed by the laboratory to the appropriate General Manager and member of Corporate QA. This fax/e-mail acts as notification of the incident.

After suspension/restriction, the lab holds all reports to clients pending review. No faxing, mailing or distributing through electronic means may occur. The report must not be posted for viewing on the internet. It is the responsibility of the Laboratory Director to hold all reporting and to notify all relevant laboratory personnel regarding the suspension/restriction (i. e., Project Management, Log-in, etc...). Clients are NOT generally notified at this time. Analysis may proceed in some instances depending on the non-conformance issue.

Within 72 hours, the QA Manager determines if compliance is now met and reports can be released, OR determine the plan of action to bring work into compliance, and release work. A team, with all principals involved (Laboratory Director, Technical Director, QA Manager, Department Supervisor) can devise a start-up plan to cover all steps from client notification through compliance and release of reports. Project Management and the Director of Client Services and Sales and Marketing are notified if clients must be notified or if the suspension/restriction affects the laboratory's ability to accept work. The Laboratory Director, with the approval of the QA Manager, has the responsibility to authorize the resumption of work or the elimination of any restrictions after all corrective action is complete. Approval is given by final signature on the completed corrective action report as described in Section 13.

SECTION 13

CORRECTIVE ACTION
(NELAC 5.4.10)

13.1 OVERVIEW

A major component of TestAmerica's Quality Assurance (QA) Program is the problem investigation and feedback mechanism designed to keep the laboratory staff informed on quality related issues and to provide insight to problem resolution. When nonconforming work or departures from policies and procedures in the quality system or technical operations are identified, the corrective action procedure provides a systematic approach to assess the issues, restore the laboratory's system integrity, and prevent reoccurrence. Corrective actions are documented using Non-Conformance Reports (NCR) and Corrective Action Reports (CAR).

13.2 GENERAL

Problems within the quality system or within analytical operations may be discovered in a variety of ways, such as QC sample failures, internal or external audits, proficiency testing (PT) performance, client complaints, staff observation, etc.

The purpose of a corrective action system is to:

- Identify non-conformance events and assign responsibility for investigation.
- Resolve non-conformance events and assign responsibility for any required corrective action.
- Identify systematic problems before they become serious.
- Identify and track client complaints and provide resolution (see more on client complaints in Section 11).

13.2.1 Non-Conformance Report (NCR) - is used to document the following types of corrective actions:

- Deviations from an established procedure or SOP (See Figure 13-1 for an example Analysis Non-conformance Report.)
- QC outside of limits (non matrix related)
- Isolated reporting / calculation errors
- Client complaints (See Figure 11-1 for an example Client Non-conformance Report.)
- Sample receipt non-conformances (See Figure 13-2 for an example Sample Non-conformance Report.)

13.2.2 Corrective Action Report (CAR) - is used to document (see Figure 13-3 for an example) the following types of corrective actions:

- Questionable trends that are found in the monthly review of NCRs.
- Issues found while reviewing NCRs that warrant further investigation.
- Internal and external audit findings
- Failed or unacceptable PT results.

- Corrective actions that cross multiple departments in the laboratory.
- Systematic reporting / calculation errors
- Health and safety violations

13.3 CLOSED LOOP CORRECTIVE ACTION PROCESS

Any employee in the company can initiate a corrective action. There are four main components to a closed-loop corrective action process once an issue has been identified: Root Cause Analysis, Selection and Implementation of Corrective Actions (both short and long term), Monitoring of the Corrective Actions, and Follow-up.

13.3.1 Root Cause Analysis

- Upon discovery of a non-conformance event, the event must be defined and documented. An NCR or CAR must be initiated, someone is assigned to investigate the issue and the event is investigated for root cause. Table 13-1 provides some general guidelines on determining responsibility for assessment.
- Root Cause Analysis is an investigative process aimed at identifying the basic or causal factor(s) that underlies variation in performance or the occurrence of a documented non-conformance event. The root cause may be buried under seemingly innocuous events, many steps preceding the perceived failure. At first glance, an immediate response is often directed at a symptom and not the cause.
- Systematically analyze and document the Root Cause(s) behind the issue. Attempt to identify Root Cause that, when corrected, potentially will lead to dramatic improvements in performance by eliminating entire classes of problems.
- Identify and review each issue associated with the event and ask why this event occurred. Brainstorm the root cause(s) of problems by asking why events occurred or conditions existed. Look at technique, processes, or other systems outside of the normal/obvious indicators. Often creative thinking will find root causes that ordinarily would be missed, and continue to plague the laboratory or operation. If there are sub issues, repeat the process for all issues associated with the non-conformance event.
- The root cause analysis step is the key to the corrective action process, as a long term corrective action cannot be determined until the root cause is determined.
- If the root cause is not readily obvious, the Department Supervisor, Operations Manager, Technical Director, Lab Director, or QA Manager (or QA designee) is consulted.

13.3.2 Selection and Implementation of Corrective Actions

- Where corrective action is needed, the laboratory must identify potential corrective actions. The action(s) most likely to address root causes, eliminate the problem, and prevent recurrence are selected and implemented. Responsibility for implementation is assigned.
- Corrective actions must be to a degree appropriate to the magnitude of the problem identified through the root cause analysis.
- Whatever corrective action is determined to be appropriate, the laboratory must document and implement the changes. The NCR or CAR is used for this documentation.

13.3.3 Monitoring of the Corrective Actions

- The Department Manager/Supervisor and QA Manager are responsible to ensure that the corrective action taken was effective.
- All Corrective action follow up is documented in the CAR database and ineffective actions are re-evaluated until acceptable resolution is achieved. Department Managers are accountable to the Laboratory Director to ensure final acceptable resolution is achieved and documented appropriately.
- Each NCR and CAR is entered into a database for tracking purposes, and a monthly summary of all corrective actions is printed out for review to aid in ensuring that the corrective actions have taken effect.
- The QA Manager reviews monthly NCRs and CARs for trends. Highlights are included in the QA monthly report (refer to Section 17). If a significant trend develops that adversely affects quality, an audit of the area is performed and corrective action implemented.
- Any out-of-control situations that are not addressed acceptably at the laboratory level may be reported to the Corporate Quality Director by the laboratory QA Manager indicating the nature of the out-of-control situation and problems encountered in solving the situation.

13.3.4 Follow-up Audits

- Follow-up audits may be initiated by the QA Manager and are performed as soon as possible when the identification of a nonconformance casts doubt on the laboratory's compliance with its own policies and procedures, or on its compliance with state or federal requirements. (Refer to Section 16.1.4 Special Audits.)
- These audits often follow the implementation of the corrective actions to verify effectiveness. An additional audit would only be necessary when a critical issue or risk to business is discovered.

13.4 TECHNICAL CORRECTIVE ACTIONS

In addition to providing acceptance criteria and specific protocols for technical corrective actions in the method SOPs, the laboratory has general procedures to be followed to determine when departures from the documented policies and procedures and quality control have occurred (refer to Section 12 for information regarding the control of non-conforming work). The documentation of these procedures is through the use of an NCR or CAR.

Table 13-1 includes examples of general technical corrective actions. For specific criteria and corrective actions refer to the analytical methods or specific method SOPs.

Table 13-1 provides some general guidelines for identifying the individual(s) responsible for assessing each QC type and initiating corrective action. The table also provides general guidance on how a data set must be treated if associated QC measurements are unacceptable. Specific procedures are included in method SOPs, QAM Sections 20 and 21, and SOP CA-L-S-001 (Internal Investigation of Potential Data Discrepancies and Determination for Data Recall. All corrective actions are reviewed monthly, at a minimum, by the QA Manager and highlights are included in the QA monthly report.

To the extent possible, samples are reported only if all quality control measures are acceptable. If the deficiency does not impair the usability of the results, data must be reported with an appropriate data qualifier and/or the deficiency must be noted in the case narrative. Where sample results may be impaired, the Project Manager is notified by an NCR and appropriate corrective action (e. g., reanalysis) is taken and documented.

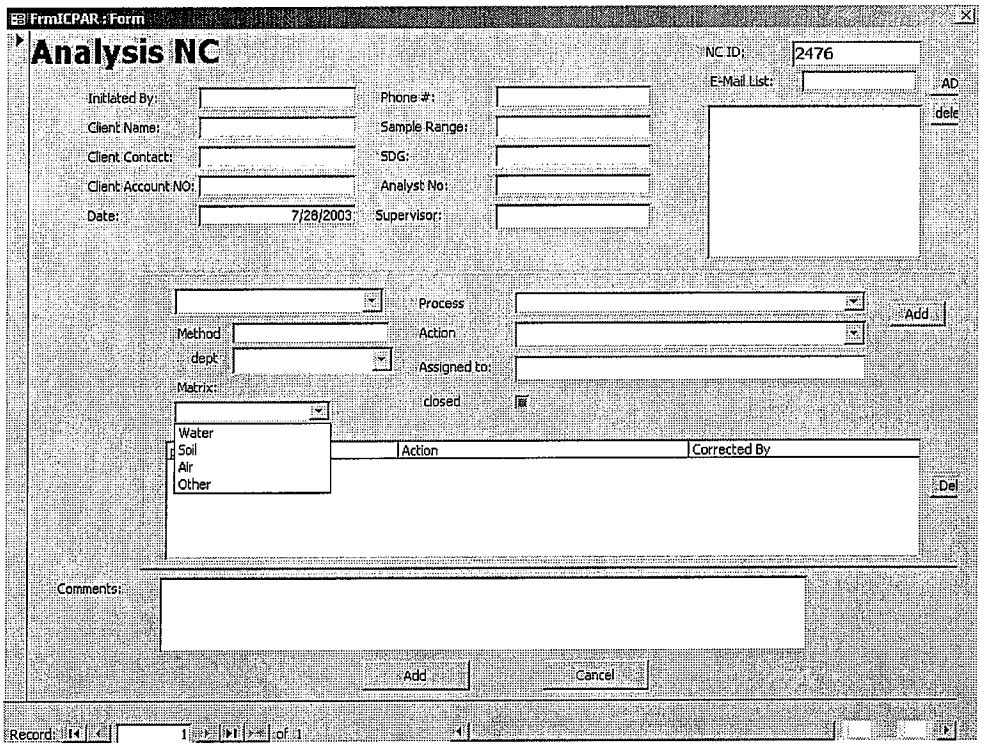
13.5 BASIC CORRECTIONS

When mistakes occur in records, each mistake must be crossed-out, and not erased, deleted, made illegible, or otherwise obliterated (e. g., no white-out), and the correct value entered alongside. All such corrections must be initialed (or signed) and dated by the person making the correction. In the case of records stored electronically, the original "uncorrected" file must be maintained intact and a second "corrected" file is created.

This same process applies to adding additional information to a record. All additions made later than the initial must also be initialed (or signed) and dated.

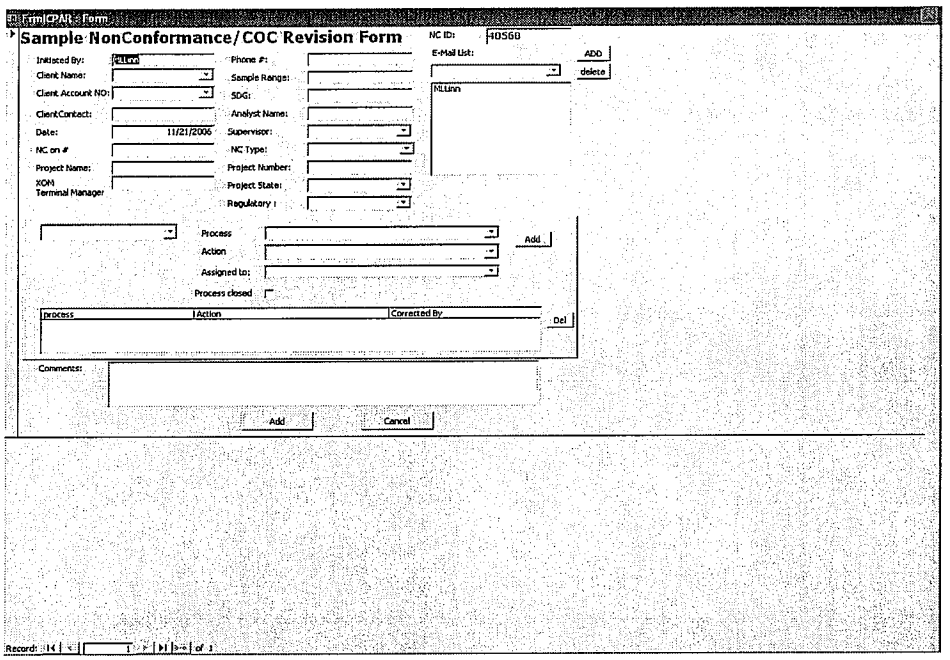
When corrections are due to reasons other than obvious transcription errors, the reason for the corrections (or additions) must also be documented.

Figure 13-1. Analysis Non-Conformance Report



First Drop-down Menu	PEST
CCV	HPLC
Ext. Blank	QC HOLD
Final Review Log-in	INORGANICS
Final Review Sample	**Process Drop-down Menu**
INST. Blank	(Describe in text)
LCS	**Action Drop-down Menu**
Log-in	Corrected action not chosen
MS/MSD	Corrective and Preventive Action Report Generated
Non Analysis	Incorrect Extraction/Extract Volumes Entered into LIMS
Other	Metals MS/MSD out. Post-digestion Spike Analyzed.
Sample Issues	Pesticide Breakdown Criteria Failed
Surrogate	Re-analyze
Dept. Drop-down Menu	Re-analyze – Out of Holding Time
BNA	Re-analyze Insufficient Sample
BTX	Re-analyze – NO (Comments needed in LIMS)
DRO	Re-analyze – NO (Comments needed in LIMS)
HALL	Re-extract – Insufficient Sample
METALS	Re-extract – Out of Holding Time
MISC	Re-extract
PREP	Sample Dilutions Did Not Confirm Original Analysis
TPH PREP	Worklist Taken Back to "Not Entered" Due to Data Entry, Unload or Analysis Error
VOA	

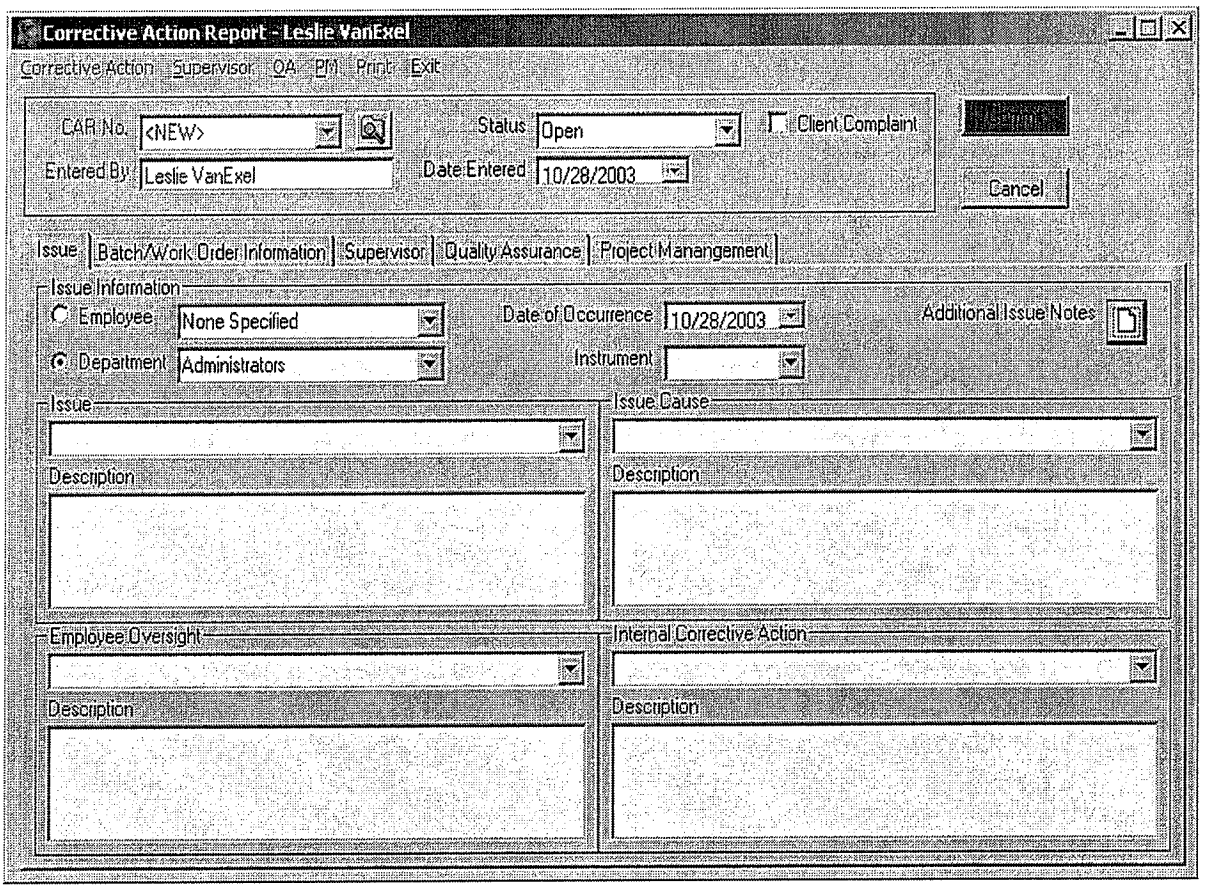
Figure 13-2. Sample Non-conformance Report

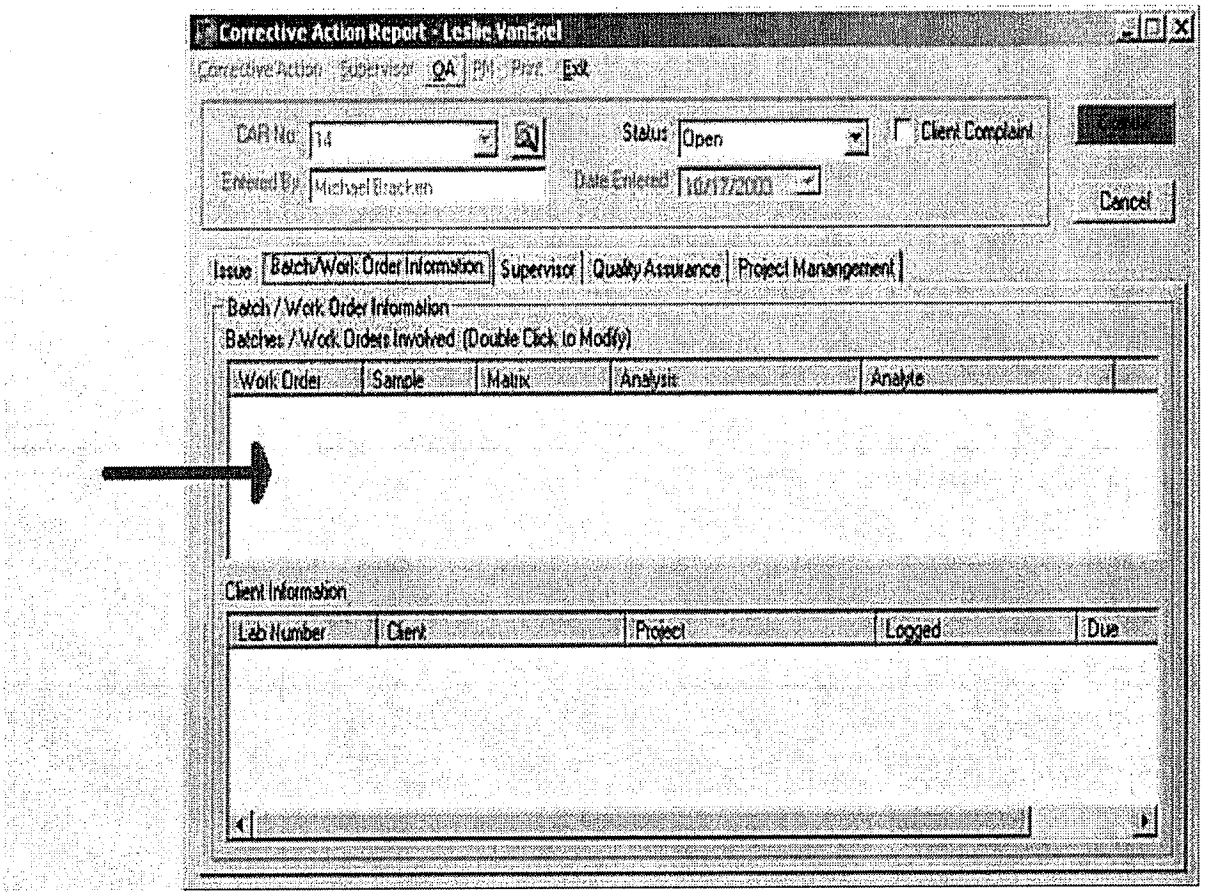


Process Drop-down Menu	Action Drop-down Menu
Change Project Name	Account Number Provided in Comments
Change Project Number	Analysis cancelled per client request
Change Sample Collection Date/Time	Analytical Method Number provided in Comments
Change Sample ID	Cancelled Analysis
Change TAT to RUSH TAT per client request	Changed TAT per client request
Change TAT to Standard TAT per client request	Chromatograms have been requested
Change/Verify Method	Clarifications of special Instructions/COC provided by client
Changed Report Units – per client notes	Client Notified
Changed Report Units – per client request	Client Notified and Cancelled Analysis
Client cancelled sample analysis after analysis performed	COC sent in by client (via fax or e-mail)
Client reimbursement requested	Compounds removed as requested
COC is unclear-please clarify	Corrected action not chosen
Containers submitted-no analysis requested on COC	Data Qualifier added/removed as requested
Correct units not used in report/LIMS	Do Not Run
Dioxin/Furans List/Method?	Documentation Level: ½
Documentation Level?	Documentation Level: 3 (Full Doc minus Raw Data)
EDB Method?	Documentation Level: 4 (Full Doc plus Raw Data)
EnCore Prep Date Incorrectly Entered in LIMS	Documentation Level: CLP Methods
Fedex Delivery Failure	EDB Method: 504.1
HERB List?	EDB Method: 524/6210
Hold sample(s) per client/PM request	EDB Method: 601
Improper Bottle Used for Analysis	EDB Method: 624
Metals List?	EDB Method: 8011
Need List of Compounds?	EDB Method: 8021
Need MA MCP/CAM Checklist Provided	EDB Method: 8260
No Analysis Requested?	Herb List: Appendix IX
No COC-Please FAX	Herb List: Long
Oil & Grease Method?	Herb List: Other
Other NC/Process: See Comment Section Below	Herb List: Short

Out of Holding Time-List Analysis	Letter has been sent to NC per state cert. requirement
PO#	List provided by client
Provide Chromatograms per client request	MA MCP/CAM Checklist Provided by Project Management
Purchase Order Number Not provided	Metals List: 8 RCRA Metals
Remove compounds not requested	Metals List: CPL/TAL
Remove compounds requested on COC	Metals List: Other
Remove or add data qualifier flags	Metals List: Priority Pollutant List
Retag for additional analysis (Sample already completed)	Need List of Compounds in Comments
Sample Containers Broken in Shipment	New Analysis added per Client Request
Sample Containers Missing from Cooler-checked twice	No
Sample mislabeled	
Samples missing that are listed on the COC	
Samples submitted with Expired Holding Time	
Saturday Delivery Marked? Yes or No	Oil & Grease Method: 9070A/9070B/1664
Service Order Number not provided	Proceed with analysis and note in comment section of rpt
Special Instructions unclear-please clarify	Process Completed
Subcontract Samples?	Purchase Order Number provided in Comment Section
TCLP?	Resampling Request Form completed w/ documentation
Temperature outside Method Allowance-Run or Do Not Run?	Run
TPH Method?	Run Method 9071B HEM (soil)
Trip Blank not located in sample cooler	Sample held as requested
Update Demographics in Database-State, Matrix, Date, etc.	Sample Placed on Hold
Verify analysis/method/compound requested	Samples subcontracted to client approved subcontractor
Write letter to NC State for Nonconformance issue	Samples retagged; New SDB in Comment Section below
Wrong Preservation Used	Service Order Number provided in Comment Section
	Void NC
	Yes

Figure 13-3. **Corrective Action Report**




Corrective Action Report - Leslie VanExel
Corrective Action
Supervisor
QA
PM
Print
Exit
CAR No
14
Status
Open
Client Complaint
Entered By
Michael Bracken
Date Entered
Cancel
Issue
Batch/Work Order Information
Supervisor
Quality Assurance
Project Management
Batch / Work Order Information
Batches / Work Orders Involved (Double Click to Modify)
Work Order
Sample
Matrix
Analysis
Analyte
Client Information
Lab Number
Client
Project
Logged
Due

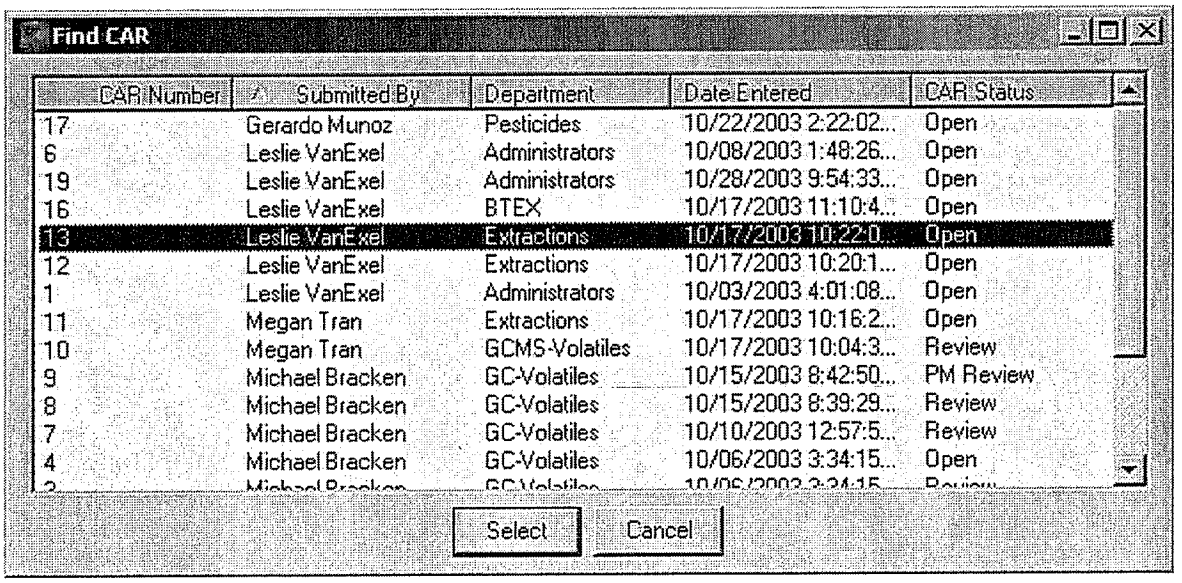

Find CAR
CAR Number
Submitted By
Department
Date Entered
CAR Status
17 Gerardo Munoz Pesticides 10/22/2003 2:22:02... Open
6 Leslie VanExel Administrators 10/08/2003 1:48:26... Open
19 Leslie VanExel Administrators 10/28/2003 9:54:33... Open
16 Leslie VanExel BTEX 10/17/2003 11:10:4... Open
13 Leslie VanExel Extractions 10/17/2003 10:22:0... Open
12 Leslie VanExel Extractions 10/17/2003 10:20:1... Open
1 Leslie VanExel Administrators 10/03/2003 4:01:08... Open
11 Megan Tran Extractions 10/17/2003 10:16:2... Open
10 Megan Tran GCMS-Volatiles 10/17/2003 10:04:3... Review
9 Michael Bracken GC-Volatiles 10/15/2003 8:42:50... PM Review
8 Michael Bracken GC-Volatiles 10/15/2003 8:39:29... Review
7 Michael Bracken GC-Volatiles 10/10/2003 12:57:5... Review
4 Michael Bracken GC-Volatiles 10/06/2003 3:34:15... Open
Select
Cancel

Table 13-1. Example – General Corrective Action Procedures

(For Ohio VAP, see Ohio specific SOPs)

QC Activity *(Individual Responsible for Initiation/Assessment)*	Acceptance Criteria	Recommended Corrective Action
Initial Instrument Blank *(Analyst)*	Instrument response < MDL.	Prepare another blank. If same response, determine cause of contamination: reagents, environment, instrument equipment failure, etc.
Initial Calibration Standards *(Analyst, Department Supervisor)*	Correlation coefficient $r \geq 0.995$ ($r^2 \geq 0.99$) or standard concentration value. % Recovery within acceptance range. See details in method SOP.	Reanalyze standards. If still unacceptable, remake standards and recalibrate instrument.
Independent Calibration Verification (Second Source) *(Analyst, Department Supervisor)*	% Recovery within control limits.	Remake and reanalyze standard. If still unacceptable, then remake calibration standards or use new primary standards and recalibrate instrument.
Continuing Calibration Standards *(Analyst, Data Reviewer)*	% Recovery within control limits.	Reanalyze standard. If still unacceptable, then recalibrate and rerun affected samples.
Matrix Spike / Matrix Spike Duplicate (MS/MSD) *(Analyst, Data Reviewer)*	% Recovery within limits documented in the Control Limits Manual	If the acceptance criteria for duplicates or matrix spikes are not met because of matrix interferences, the acceptance of the analytical batch is determined by the validity of the LCS. If the LCS is within acceptable limits, the batch is acceptable. The results of the duplicates, matrix spikes and the LCS are reported with the data set.
Laboratory Control Sample (LCS) *(Analyst, Data Reviewer)*	% Recovery within limits specified in the Control Limits Manual	Batch must be re-prepared and re-analyzed. If there is insufficient sample or the holding time cannot be met, contact client and report with flags.
Laboratory Control Sample Duplicate (LCSD) *(Analyst, Data Reviewer)*	% Recovery within limits specified in the Control Limits Manual	Batch must be re-prepared and re-analyzed. If there is insufficient sample or the holding time cannot be met, contact client and report with flags.
Surrogates in environmental samples *(Analyst, Data Reviewer)*	% Recovery within limits of method or within three standard deviations of the historical mean.	Individual sample must be repeated. Place comment in LIMS.

QC Activity *(Individual Responsible for Initiation/Assessment)*	Acceptance Criteria	Recommended Corrective Action
Surrogates in batch QC samples *(Analyst, Data Reviewer)*	Within limits of method or within three standard deviations of the historical mean.	Individual sample must be repeated. Place comment in LIMS. The batch may require repreparation and/or analysis if the QC sample calls into question the acceptability of the samples. If the surrogate % recovery exceeds the upper control limit AND any associated client samples in the batch are non-detect for target compounds, the data is acceptable to report. If the surrogate % recovery exceeds the upper control limit AND any associated client samples are greater than the RL, re-prep and re-analyze affected QC and client samples in the batch.
Method Blank (MB_ *(Analyst, Data Reviewer)*	< Reporting Limit [1]	Reanalyze blank. If still positive, determine source of contamination. If necessary, reprocess (i. e., digest or extract) entire sample batch. Report blank results.
Proficiency Testing (PT) Samples *(QA Manager, Department Supervisor)*	Criteria supplied by PT Supplier.	Any failures or warnings must be investigated for cause. Failures may result in the need to repeat a PT sample to show the problem is corrected.
Internal / External Audits *(QA Manager, Department Supervisor, Operations Manager, Technical Director, Laboratory Director)*	Defined in Quality System documentation such as SOPs, QAM, etc.	Non-conformances must be investigated through CAR system and necessary corrections must be made.
Reporting / Calculation Errors (Depends on issue – possible individuals include: Analysts, Data Reviewers, Project Managers, Department Supervisor, QA *Manager*, Corporate QA, Corporate Management)	SOP CA-L-S-001, Internal Investigation of Potential Data Discrepancies and Determination for Data Recall.	Corrective action is determined by type of error. Follow the procedures in SOP CA-L-S-001.

QC Activity *(Individual Responsible for Initiation/Assessment)*	Acceptance Criteria	Recommended Corrective Action
Client Complaints *(Project Managers, Lab Director, Sales and Marketing)*		Corrective action is determined by the type of complaint. For example, a complaint regarding an incorrect address on a report must result in the report being corrected and then follow-up must be performed on the reasons the address was incorrect (e.g., database needs to be updated).
QA Monthly Report (Refer to Section 17 for an example) (QA *Manager*, Lab Director, Department Supervisors/Managers)	QAM, SOPs.	Corrective action is determined by the type of issue. For example, CARs for the month are reviewed and possible trends are investigated.
Health and Safety Violation (Health & Safety Officer, Lab Director, Department Supervisor)	Environmental Health and Safety (EHS) Manual.	Non-conformance is investigated and corrected through CAR system.

Note:
1. Except as noted below for certain compounds, the method blank must be below the detection limit. Concentrations up to five times the reporting limit are allowed for the ubiquitous laboratory and reagent contaminants: methylene chloride, toluene, acetone, 2-butanone and phthalates **provided** they appear in similar levels in the reagent blank and samples. This allowance presumes that the detection limit is significantly below any regulatory limit to which the data are to be compared and that blank subtraction is not performed. For benzene and ethylene dibromide (EDB) and other analytes for which regulatory limits are extremely close to the detection limit, the method blank must be below the method detection limit. For Ohio VAP, the method blank must be below the reporting limit for all analytes that are a contaminant of concern for the project site.

SECTION 14

PREVENTIVE ACTION
(NELAC 5.4.11)

14.1 OVERVIEW

The laboratory's preventive action programs improve, or eliminate potential causes of nonconforming product and/or nonconformance to the quality system. This preventive action process is a proactive continuous process improvement activity that can be initiated through feedback from clients, employees, business providers, and affiliates. The QA Department has the overall responsibility to ensure that the preventive action process is in place, and that relevant information on actions is submitted for management review.

Dedicating resources to an effective preventive action system emphasizes TestAmerica Nashville's commitment to its Quality Program. It is beneficial to identify and address negative trends before they develop into complaints, problems and corrective actions. Additionally, customer service and satisfaction can be improved through continuous improvements to laboratory systems.

Opportunities for improvement may be discovered during management reviews, the QA Metrics Report, internal or external audits, proficiency testing performance, client complaints, staff observation, etc.

The monthly QA Metrics Report shows performance indicators in all areas of the quality system. These areas include revised reports, corrective actions, audit findings, internal auditing and data authenticity audits, client complaints, PT samples, holding time violations, SOPs, ethics training, etc. These metrics are used to help evaluate quality system performance on an ongoing basis and provide a tool for identifying areas for improvement.

The laboratory's corrective action process (Section 13) is integral to implementation of preventive actions. A critical piece of the corrective action process is the implementation of actions to prevent further occurrence of a non-compliance event. Historical review of corrective action provides a valuable mechanism for identifying preventive action opportunities.

14.1.1 The following elements are part of a preventive action system:

- Identification of an opportunity for preventive action.
- Process for the preventive action.
- Define the measurements of the effectiveness of the process once undertaken.
- Execution of the preventive action.
- Evaluation of the plan using the defined measurements.
- Verification of the effectiveness of the preventive action.
- Close-Out by documenting any permanent changes to the Quality System as a result of the Preventive Action. Documentation of Preventive Action is incorporated into the monthly QA reports, corrective action process, management reviews, and the Management of Change process (see below).

Note: There may be varying levels of formality and documentation during the preventive action process due to the simplicity/complexity of the action taken.

14.1.2 Any Preventive Actions undertaken or attempted must be taken into account during the Annual Management Review (Section 17). A highly detailed recap is not required; a simple recount of success and failure within the preventive action program provides management a measure for evaluation.

14.2 MANAGEMENT OF CHANGE

The Management of Change process is designed to manage significant events and changes that occur within the laboratory. Through these procedures, the potential risks inherent with a new event or change are identified and evaluated. The risks are minimized or eliminated through pre-planning and the development of preventive measures. The types of changes covered under this system include: Facility Changes, Major Accreditation Changes, Addition or Deletion to Division's Capabilities or Instrumentation, Key Personnel Changes, Laboratory Information Management System (LIMS) changes. This process is discussed in further detail in SOP 08-161, Management of Change.

SECTION 15

CONTROL OF RECORDS
(NELAC 5.4.12)

TestAmerica Nashville maintains a record system appropriate to its needs and that complies with applicable standards or regulations as required. The system produces unequivocal, accurate records that document all laboratory activities. The laboratory retains all original observations, calculations and derived data, calibration records and a copy of the analytical report for a minimum of five years after it has been issued. See SOP Archiving / SA08-194 for the archiving procedures.

15.1 OVERVIEW

The laboratory has established procedures for identification, collection, indexing, access, filing, storage, maintenance and disposal of quality and technical records. A record index is listed in Table 15-1. Quality records are maintained by the Quality Assurance (QA) department in a database, which is backed up as part of the regular network backup. Records are of two types; either electronic or hard copy paper formats depending on whether the record is computer or hand generated (some records may be in both formats). Technical records are maintained by the QA Department.

Table 15-1. Record Index[1]

	Record Types [1]:	Retention Time:
Technical Records	- Raw Data - Logbooks[2] - Standards - Certificates - Analytical Records - Lab Reports	5 Years from analytical report issue*
Official Documents	- Quality Assurance Manual (QAM) - Work Instructions - Policies - SOPs - Policy Memorandums - Manuals	5 Years from document retirement date*
QA Records	- Internal & External Audits/Respones - Certifications - Corrective/Preventive Actions - Management Reviews - Method & Software Validation / Verification Data - Data Investigation	5 Years from archival* **Data Investigation:** 5 years or the life of the affected raw data storage whichever is greater (beyond 5 years if ongoing project or pending investigation)

	Record Types [1]:	Retention Time:
Project Records	- Sample Receipt & COC Documentation - Contracts and Amendments - Correspondence - QAPP -SAP - Telephone Logbooks - Lab Reports	5 Years from analytical report issue*
Administrative Records	Finance and Accounting	10 years
	EH&S Manual, Permits, Disposal Records	7 years
	Employee Handbook	Indefinitely
	Personnel files, Employee Signature & Initials, Administrative Training Records (e.g., Ethics)	7 Years (HR Personnel Files must be maintained indefinitely)
	Administrative Policies Technical Training Records	7 years

[1] Record Types encompass hardcopy and electronic records.
[2] Examples of Logbook types: Maintenance, Instrument Run, Preparation (standard and samples), Standard and Reagent Receipt, Archiving, Balance Calibration, Temperature (hardcopy or electronic records).
* Exceptions listed in Table 15-2.

All records are legible and stored and retained in such a way that they are secure and readily retrievable at the laboratory facility or an offsite location that provides a suitable environment to prevent damage or deterioration and to prevent loss at the Security Archives data storage facility. All records are protected against fire, theft, loss, environmental deterioration, and vermin. In the case of electronic records, electronic or magnetic sources, storage media are protected from deterioration caused by magnetic fields and/or electronic deterioration. Records are maintained on-site at the laboratory for at least one year after their generation and moved offsite for the remainder of the required storage time. Records are maintained for a minimum of five years unless otherwise specified by a client or regulatory requirement.

For raw data and project records, record retention is calculated from the date the project report is issued. For other records, such as Controlled Documents, QA, or Administrative Records, the retention time is calculated from the date the record is formally retired. Records related to the programs listed in Table 15-2 have lengthier retention requirements and are subject to the requirements in Section 15.1.3.

15.1.1 Programs with Longer Retention Requirements

Some regulatory programs have longer record retention requirements than the standard record retention time. These are detailed in Table 15-2 with their retention requirements. In these cases, the longer retention requirement is enacted. If special instructions exist such that client data cannot be destroyed prior to notification of the client, the container or box containing that data is marked as to whom to contact for authorization prior to destroying the data.

Table 15-2. Special Record Retention Requirements

Program	[1]Retention Requirement
Drinking Water – All States	10 years (project records)
Drinking Water Lead and Copper Rule	12 years (project records)
Commonwealth of MA – All environmental data 310 CMR 42.14	10 years
FIFRA – 40 CFR Part 160	Retain for life of research or marketing permit for pesticides regulated by EPA
Housing and Urban Development (HUD) Environmental Lead Testing	10 years
Alaska	10 years
Louisiana – All	10 years
Michigan Department of Environmental Quality – all environmental data	10 years
Navy Facilities Engineering Service Center (NFESC)	10 years
NY Potable Water NYCRR Part 55-2	10 years
OH VAP	10 years and State contacted prior to disposal
TSCA - 40 CFR Part 792	10 years after publication of final test rule or negotiated test agreement

[1]Note: Extended retention requirements must be noted with the archive documents or addressed in facility-specific records retention procedures.

15.1.2 All records are held secure and in confidence. Records maintained at the laboratory are located in the Data Archive Room. Records archived off-site are stored in a secure location where a record is maintained of any entry into the storage facility. Logs are maintained in each storage box to note removal and return of records.

15.1.3 The laboratory has procedures to protect and back-up records stored electronically and to prevent unauthorized access to or amendment of these records. All analytical data is maintained as hard copy or in a secure readable electronic format. For analytical reports that are maintained as copies in PDF format, see Section 20.12.1, "Computer and Electronic Data Related Requirements" for more information. Also reference SOP Electronic Lab Data Archiving / NV08-171.

15.1.4 The record keeping system allows for historical reconstruction of all laboratory activities that produced the analytical data, as well as rapid recovery of historical data. The history of the sample from when the laboratory took possession must be readily understood through the documentation. This includes inter-laboratory transfers of samples and/or extracts.

- The records include the identity of personnel involved in sampling, sample receipt, preparation, or testing. All analytical work contains the initials (at least) of the personnel involved. The laboratory's copy of the chain of custody is stored with the project folder. An electronic copy is stored on the server. The chain of custody indicates the name of the

sampler. If any sampling notes are provided with a work order, they are kept with this package.

- All information relating to the laboratory facilities equipment, analytical test methods, and related laboratory activities, such as sample receipt, sample preparation, or data verification are documented. The LIMS maintains an audit trail of data verification steps.

- The record keeping system facilitates the retrieval of all working files and archived records for inspection and verification purposes (e. g., set format for naming electronic files, set format for what is included with a given analytical data set). See SOP Electronic Lab Data Archiving / NV08-171. At a minimum, a data folder includes: benchsheet, prep sheets (where applicable), sample data, and associated QC data. Analytical data is stored by department and sequentially by date analyzed. Run logs are maintained for each instrument or method; a copy of each day's run long or instrument sequence is stored with the data to aid in reconstructing an analytical sequence. Where an analysis is performed without an instrument, bound logbooks or bench sheets are used to record and file data. Standard and reagent information is recorded in logbooks or entered into the LIMS for each method as required. Each folder containing a batch of data is labeled, at a minimum, with the date analyzed and the benchsheet or sequence number.

- Changes to hardcopy records must follow the procedures outlined in Sections 13 and 20. Changes to electronic records in LIMS or instrument data are recorded in audit trails.

- The reason for a signature or initials on a document is clearly indicated in the records such as "sampled by," "prepared by," "reviewed by", or "Analyzed by."

- All generated data, except those that are generated by automated data collection systems, are recorded directly, promptly and legibly in permanent dark ink.

- Hard copy data may be scanned into PDF format for record storage. Prior to the destruction of any of the hard copy information that was scanned, the scanning process must be verified in order to ensure that no data is lost, and the data files and storage media must be tested to verify the laboratory's ability to retrieve the information.

- Also refer to Section 20.13.1, "Computer and Electronic Data Related Requirements."

15.2 TECHNICAL AND ANALYTICAL RECORDS

15.2.1 The laboratory retains records of original observations, derived data and sufficient information to establish an audit trail, calibration records, staff records and a copy of each analytical report issued, for a minimum of five years unless otherwise specified by a client or regulatory requirement (refer to Section 15.1). The records for each analysis must contain sufficient information to enable the analysis to be repeated under conditions as close as possible to the original. The records must include the identity of laboratory personnel responsible for the sampling, performance of each analysis and reviewing of results.

15.2.2 Observations, data and calculations are recorded at the time they are made and are identifiable to the specific task.

15.2.3 Changes to hardcopy records must follow the procedures outlined in Section 13 and 20. Changes to electronic records in LIMS or instrument data are recorded in audit trails.

15.2.4 The essential information to be associated with analysis, such as strip charts, tabular printouts, computer data files, analytical notebooks, and run logs, include:

- laboratory sample ID code;
- date of analysis; time of analysis is also required if the holding time is seventy-two (72) hours or less, or when time critical steps are included in the analysis (e.g., drying times, incubations, etc.); instrumental analyses have the date and time of analysis recorded as part of their general operations. Where a time critical step exists in an analysis, location for such a time is included as part of the documentation in a specific logbook or on a benchsheet.
- instrumentation identification and instrument operating conditions/parameters. Operating conditions/parameters are recorded in instrument maintenance logs where available.
- analysis type;
- all manual calculations and manual integrations;
- analyst's or operator's initials/signature;
- sample preparation including cleanup, separation protocols, incubation periods, ID codes, volumes, weights, instrument printouts, meter readings, calculations, reagents;
- test results;
- standard and reagent origin, receipt, preparation, and use;
- calibration criteria, frequency and acceptance criteria;
- data and statistical calculations, review, confirmation, interpretation, assessment and reporting conventions;
- quality control protocols and assessment;
- electronic data security, software documentation and verification, software and hardware audits, backups, and records of any changes to automated data entries; and
- method performance criteria including expected quality control requirements. These are indicated both in the LIMS and on specific analytical report formats.

15.3 LABORATORY SUPPORT ACTIVITIES

In addition to documenting all the above-mentioned activities, the following are retained QA records and project records (previous discussions in this section relate where and how these data are stored):

- all original raw data, whether hard copy or electronic, for calibrations, samples and quality control measures, including analysts' work sheets and data output records (chromatograms, strip charts, and other instrument response readout records);
- a written description or reference to the specific test method used which includes a description of the specific computational steps used to translate parametric observations into a reportable analytical value;
- copies of final reports;

- archived SOPs;
- correspondence relating to laboratory activities for a specific project;
- all corrective action reports, audits and audit responses;
- proficiency test results and raw data; and
- results of data review, verification, and crosschecking procedures

15.3.1 Sample Handling Records

Sample handling and tracking is discussed in Section 24. Records of all procedures to which a sample is subjected while in the possession of the laboratory are maintained. These include but are not limited to records pertaining to:

- sample preservation including appropriateness of sample container and compliance with holding time requirement;
- sample identification, receipt, acceptance or rejection and login;
- sample storage and tracking including shipping receipts, sample transmittal / COC forms; and
- procedures for the receipt and retention of samples, including all provisions necessary to protect the integrity of samples.

15.4 ADMINISTRATIVE RECORDS

The laboratory also maintains the administrative records in either electronic or hard copy form. See Table 15-1.

15.5 RECORDS MANAGEMENT, STORAGE AND DISPOSAL

15.5.1 All records (including those pertaining to test equipment), certificates and reports are safely stored, held secure and in confidence to the client. Certification related records are available upon request.

15.5.2 All information necessary for the historical reconstruction of data is maintained by the laboratory. Records that are stored only on electronic media must be supported by the hardware and software necessary for their retrieval.

15.5.3 Records that are stored or generated by computers or personal computers have hard copy, write-protected backup copies, or an electronic audit trail controlling access.

15.5.4 TestAmerica Nashville has a record management system (a.k.a document control) for control of laboratory notebooks, instrument logbooks, standards logbooks, and records for data reduction, validation, storage and reporting. Laboratory notebooks are issued on a per analysis basis and are numbered sequentially within a given analysis. No analysis has more than one active notebook at a time, so all data are recorded sequentially within a series of sequential notebooks. Bench sheets are filed sequentially in folders. Standards are maintained in the LIMS; no logbooks are used to record that data.

15.5.5 Records are considered archived when moved off-site. Access to archived hard-copy information is documented with an access log and in/out records is used in archived boxes to note data that is removed and returned. Access to the data is limited to data storage facility and TestAmerica employees.

15.5.6 Transfer of Ownership

15.5.6.1 In the event that the laboratory transfers ownership or goes out of business, TestAmerica Nashville must ensure that the records are maintained or transferred according to client's instructions. Upon ownership transfer, record retention requirements must be addressed in the ownership transfer agreement and the responsibility for maintaining archives is clearly established. In addition, in cases of bankruptcy, appropriate regulatory and state legal requirements concerning laboratory records must be followed. In the event of the closure of the laboratory, all records must revert to the control of the corporate headquarters. If the entire company cease to exist, as much notice as possible must be given to clients and the accrediting bodies who have worked with the laboratory during the previous 5 years of such action.

15.5.7 Records Disposal

15.5.7.1 Records are removed from the archive and destroyed after 10 years unless otherwise specified by a client or regulatory requirement. On a project specific or program basis, clients may need to be notified prior to record destruction. Records are destroyed in a manner that ensures their confidentiality such as shredding, mutilation or incineration.

15.5.7.2 Electronic copies of records must be destroyed by erasure or physically damaging off-line storage media so no records can be read.

15.5.7.3 If a third party records management company is hired to dispose of records, a "Certificate of Destruction" is required.

SECTION 16

AUDITS
(NELAC 5.4.13)

16.1 INTERNAL AUDITS

Internal audits are performed to verify that laboratory operations comply with the requirements of the lab's quality system and with the external quality programs under which the laboratory operates. Audits are planned and organized by the QA staff. Personnel conducting the audits should be independent of the area being evaluated. Auditors have the sufficient authority, access to work areas, and organizational freedom necessary to observe all activities affecting quality and to report the assessments to laboratory management and when requested to corporate management.

Audits are conducted and documented as described in the TestAmerica Corporate SOP on performing Internal Audits, SOP No. CA-Q-S-004. The types and frequency of routine internal audits are shown in Table 16-1. Special or ad hoc assessments may be conducted as needed under the direction of the QA staff.

Table 16-1. Types of Internal Audits and Frequency

Description	Performed by	Frequency
Quality Systems	QA Department or Designee	All areas of the laboratory annually
QA Technical Audits - Evaluate raw data versus final reports - Analyst integrity - Data authenticity	QA Department or Designee	All methods within a 2-year period, with at least 15% of methods every quarter
SOP Method Compliance	Technical Director and QA Department	- All SOPs within a 2-year period - All new analysts or new analyst/methods within 3 months of IDOC
Special	QA Department or Designee	Surveillance or spot checks performed as needed
Performance Testing	Analysts with QA oversight	Two successful per year for each NELAC field of testing or as dictated by regulatory requirements

16.1.1 Annual Quality Systems Audit

An annual quality systems audit is required to ensure compliance to analytical methods and SOPs, the laboratory's Data Integrity and Ethics Policies, NELAC quality systems, ISO 17025:2005, A2LA, AIHA, client and state requirements, and the effectiveness of the internal controls of the analytical process, including but not limited to data review, quality controls, preventive action and corrective action. The completeness of earlier corrective actions is

assessed. The audit is divided into modules for each operating or support area of the lab, and each module is comprehensive for a given area. The area audits may be done on a rotating schedule throughout the year to ensure adequate coverage of all areas. This schedule may change as situations in the laboratory warrant.

16.1.2 QA Technical Audits

QA technical audits are based on client projects, associated sample delivery groups, and the methods performed. Reported results are compared to raw data to verify the authenticity of results. The validity of calibrations and QC results are compared to data qualifiers, footnotes, and case narratives. Documentation is assessed by examining run logs and records of manual integrations. Manual calculations are checked. Where possible, MintMiner is used to identify unusual manipulations of the data deserving closer scrutiny. QA technical audits must include all methods within a two-year period.

16.1.3 SOP Method Compliance

Compliance of all SOPs with the source methods and compliance of the operational groups with the SOPs must be assessed by the Technical Director and QA department at least every two years. The work of each newly hired analyst is assessed within 3 months of working independently, (e.g., completion of method IDOC). In addition, as analysts add methods to their capabilities, (new IDOC) reviews of the analyst work products must be performed within 3 months of completing the documented training.

16.1.4 Special Audits

Special audits are conducted on an as needed basis, generally as a follow up to specific issues such as client complaints, corrective actions, PT results, data audits, system audits, validation comments, regulatory audits or suspected ethical improprieties. Special audits are focused on a specific issue, and report format, distribution, and timeframes are designed to address the nature of the issue.

16.1.5 Performance Testing

The laboratory participates semi-annually in performance audits conducted through the analysis of PT samples provided by a third party. The laboratory generally participates in the following types of PT studies: drinking water, nonpotable water, soil and industrial hygiene.

It is TestAmerica's policy that PT samples be treated as typical samples in the production process. Furthermore, where PT samples present special or unique problems, in the regular production process they may need to be treated differently, as would any special or unique request submitted by any client. The QA Manager must be consulted and in agreement with any decisions made to treat a PT sample differently due to some special circumstance.

- PTs generally do not have holding times associated with them. In the absence of any holding time requirement, it is recommended that the holding time begin when the PT sample is prepared according to the manufacturers instructions. Holding times apply to full volume PT samples only if the provider gives a meaningful "sampling date." If this is not provided, it is recommended that the date/time of opening of the full volume sample be considered the beginning of holding time.

- Login obtains the COC information from the documentation provided with the PTs, with review by QA or other designated staff.

- Vials are prepared as required in the instruction set provided with the samples. After preparation to full volume, the sample is spiked, digested, concentrated, etc., as would be done for any normal sample requiring similar analysis.

- PT samples do not undergo multiple preps, multiple runs, multiple methods (unless being used to evaluate multiple methods), multiple dilutions, unless this is what would be done to a normal client sample (e. g., if a client requests, as PT clients do, that we split VOA coeluters, then dual analysis is normal practice).

- The type, composition, concentration and frequency of quality control samples analyzed with the PT samples is the same as with routine environmental samples.

- Instructions are allowed to be included in the laboratory's SOPs for how low level samples are analyzed, including concentration of the sample or adjustment of the normality of titrant. When a PT sample falls below the range of the routine analytical method, the low-level procedure would be allowed where described in the SOP.

- No special reviews are performed by operations and QA, unless this is what would be done to a normal client sample. To the degree that special report forms or login procedures are required by the PT supplier, it is reasonable that the laboratory would apply special review procedures, as would be done for any client requesting unusual reporting or login processes.

Written responses to unacceptable PT results are required. In some cases it may be necessary for blind QC samples to be submitted to the laboratory to show a return to control.

16.2 EXTERNAL AUDITS

External audits are performed when certifying agencies or clients conduct on-site inspections or submit performance testing samples for analysis. It is TestAmerica's policy to cooperate fully with regulatory authorities and clients. The laboratory makes every effort to provide the auditors with access to personnel, documentation, and assistance. Laboratory department supervisors are responsible for providing corrective actions to the QA Manager who coordinates the response for any deficiencies discovered during an external audit. Audit responses are due in the time allotted by the client or agency performing the audit.

The laboratory cooperates with clients and their representatives to monitor the laboratory's performance in relation to work performed for the client. The client may only view data and systems related directly to the client's work. All efforts are made to keep other client information confidential.

16.2.1 Confidential Business Information (CBI) Considerations

During on-site audits, auditors may come into possession of information claimed as business confidential. A business confidentiality claim is defined as "a claim or allegation that business

information is entitled to confidential treatment for reasons of business confidentiality or a request for a determination that such information is entitled to such treatment." When information is claimed as business confidential, the laboratory must place on (or attach to) the information at the time it is submitted to the auditor, a cover sheet, stamped or typed legend or other suitable form of notice, employing language such as "trade secret", "proprietary" or "company confidential". Confidential portions of documents otherwise non-confidential must be clearly identified. CBI may be purged of references to client identity by the responsible laboratory official at the time of removal from the laboratory. However, sample identifiers must not be obscured from the information. Additional information regarding CBI can be found in within the 2003 NELAC standards.

16.3 AUDIT FINDINGS

Audit findings are documented using the corrective action process and database (refer to Section 13). The laboratory's corrective action responses for both types of audits (internal and external) are completed within a predetermined timeframe. When the corrective action has not yet been completed within that predefined response timeframe, a completion date must set and agreed to by operations management and the QA Manager.

Developing and implementing corrective actions to findings is the responsibility of the Department Supervisor where the finding originated. Findings that are not corrected by specified due dates are reported monthly to management in the QA monthly report. When requested, a copy of the audit report and the labs corrective action plan are forwarded to Corporate Quality.

If any audit finding casts doubt on the effectiveness of the operations or on the correctness or validity of the laboratory's test results, the laboratory must take timely corrective action, and must notify clients in writing if the investigations show that the laboratory results have been affected. Once corrective action is implemented, a follow-up audit is scheduled to ensure that the problem has been corrected.

Clients must be notified promptly in writing, of any event such as the identification of defective measuring or test equipment that casts doubt on the validity of results given in any test report or amendment to a test report. The investigation must begin within 24-hours of discovery of the problem and all efforts are made to notify the client within two weeks after the completion of the investigation.

SECTION 17

MANAGEMENT REVIEWS
(NELAC 5.4.14)

17.1 QUALITY ASSURANCE REPORT

A comprehensive QA Report is prepared each month by the laboratory's QA Department and forwarded to the Laboratory Director for review and comments. The final report is also submitted to the Technical Director and lab Operation Managers as well as the appropriate Corporate Quality Director. All aspects of the QA system are reviewed to evaluate the suitability of policies and procedures. During the course of the year, the Laboratory Director, General Manager or Corporate QA may request that additional information be added to the report.

On a monthly basis, Corporate QA compiles information from all the monthly laboratory reports. The Corporate Quality Directors prepare a report that includes a compilation of all metrics and notable information and concerns regarding the QA programs within the laboratories. The report also includes a listing of new regulations that may potentially impact the laboratories. This report is presented to the Corporate Senior Management Team and General Managers by Corporate QA.

17.2 ANNUAL MANAGEMENT REVIEW

The senior lab management team (Laboratory Director, Technical Director, QA Manager, Operations Managers) conducts an annual review of its quality systems and LIMS to ensure its continuing suitability and effectiveness in meeting client and regulatory requirements and to introduce any necessary changes or improvements. It also provides a platform for defining quality goals & objectives.

This management systems review (Corporate SOP CA-Q-S-008 & Work Instruction No. CA-Q-WI-020) uses information generated during the preceding year to assess the "big picture" by ensuring that routine actions taken and reviewed on a monthly basis are not components of larger systematic concerns. The monthly review keeps the quality systems current and effective; therefore, the annual review is a formal senior management process to review specific existing documentation. Significant issues from the following documentation are compiled or summarized by the QA Manager prior to the review meeting:

- Matters arising from the previous annual review.
- Prior Monthly QA Reports (summarizing items, such as SOPs, CARs, MDLs, audits (internal and external), proficiency testing results, certifications, and training issues).
- Laboratory QA Metrics.
- Review of report reissue requests.
- Review of client feedback and complaints.
- Issues arising from any prior management or staff meetings.
- Minutes from prior laboratory senior management meetings. Issues that may be raised from these meetings include:

  - Adequacy of staff, equipment and facility resources.
  - Adequacy of policies and procedures.
  - Future plans for resources and testing capability and capacity.
- Review of the ACIL seal of excellence program performance.
- Compliance to the Ethics Policy and Data Integrity Plan, including any evidence/incidents of inappropriate actions or vulnerabilities related to data integrity.

The annual review includes the previous 12 months. Based on the annual review, a report is generated by the QA Manager and management. The report is distributed to the appropriate General Manager and Corporate Quality Director. The report includes, but is not limited to:

- The date of the review and the names and titles of participants.
- A reference to the existing data quality related documents and topics that were reviewed.
- Quality system or operational changes or improvements that will be made as a result of the review [e. g., an implementation schedule including assigned responsibilities for the changes (Action Table)].

The QA Manual is also reviewed at this time and revised to reflect any significant changes made to the quality systems.

17.3 POTENTIAL INTEGRITY RELATED MANAGERIAL REVIEWS

Potential integrity issues (data or business related) must be handled and reviewed in a confidential manner until such time as a follow-up evaluation, full investigation, or other appropriate actions have been completed and issues clarified. The Corporate Data Investigation/ Recall SOP must be followed (SOP No. CA-L-S-001). All investigations that result in finding of inappropriate activity are documented and include any disciplinary actions involved, corrective actions taken, and all appropriate notifications of clients.

TestAmerica's Chief Operating Officer, VP of Client and Technical Services, General Managers, and Corporate Quality Directors receive a monthly report compiled from the Director of Quality & Client Advocacy summarizing any current data integrity or data recall investigations. The General Managers are also made aware of progress on these issues for their specific labs.

SECTION 18

PERSONNEL
(NELAC 5.5.2)

18.1 OVERVIEW

The laboratory's management believes that its highly qualified and professional staff is the single most important aspect in assuring a high level of data quality and service. The staff consists of professionals and support personnel as outlined in the organization chart in Appendix 2.

All personnel must demonstrate competence in the areas where they have responsibility. Any staff member that is undergoing training must have appropriate supervision until they have demonstrated their ability to perform their job function on their own. Staff are qualified for their tasks based on appropriate education, training, experience and/or demonstrated skills as required.

The laboratory employs sufficient personnel with the necessary education, training, technical knowledge and experience for their assigned responsibilities.

All personnel are responsible for complying with all QA/QC requirements that pertain to the laboratory and their area of responsibility. Each staff member must have a combination of experience and education to adequately demonstrate a specific knowledge of their particular area of responsibility. Technical staff must also have a general knowledge of lab operations, test methods, QA/QC procedures and records management.

Laboratory management is responsible for formulating goals for lab staff with respect to education, training and skills and ensuring that the laboratory has a policy and procedures for identifying training needs and providing training of personnel. The training must be relevant to the present and anticipated responsibilities of the lab staff.

The laboratory only uses personnel that are employed by or under contract to the laboratory. Contracted personnel, when used, must meet competency standards of the laboratory and work in accordance to the laboratory's quality system.

18.2 EDUCATION AND EXPERIENCE REQUIREMENTS FOR TECHNICAL PERSONNEL

The laboratory makes every effort to hire analytical staff that possess a college degree (AA, BA, BS) in an applied science with some chemistry in the curriculum. Exceptions can be made based upon the individual's experience and ability to learn. Experience and specialized training are occasionally accepted in lieu of a college degree. (Basic lab skills such as using a balance, etc. are also considered). For any analysis, thorough training and working with another experienced staff member until proficiency has been demonstrated is required.

Selection of qualified candidates for laboratory employment begins with documentation of minimum education, training, and experience prerequisites needed to perform the prescribed task. Minimum

education and training requirements for TestAmerica employees are outlined in job descriptions and are generally summarized for analytical staff in the table below.

The laboratory maintains job descriptions for all personnel who manage, perform or verify work affecting the quality of the environmental testing the laboratory performs. Job descriptions are located on the TestAmerica intranet site's Human Resources web-page. (Also see Section 4 for position descriptions/responsibilities.)

As a general rule for analytical staff:

Specialty	Education	Experience
Extractions, Digestions, some electrode methods (pH, DO, Redox, etc.), or Titrimetric and Gravimetric Analyses	H.S. Diploma	On the job training (OJT)
CVAA, Single component or short list Chromatography (e. g., Fuels, BTEX-GC, IC)	A college degree in an applied science or 2 years of college and at least 1 year of college applied sciences	Or 2 years prior analytical experience is required
ICP, ICPMS, Long List or complex chromatography (e.g., Pesticides, PCB, Herbicides, HPLC, etc.), GCMS	A college degree in an applied science or 2 years of college applied sciences	Or 5 years of prior analytical experience
Spectra Interpretation	A college degree in an applied science or 2 years of college applied sciences	And 2 years relevant experience Or 5 years of prior analytical experience
Operations Managers/Department Supervisors	Bachelor's Degree in an applied science or engineering An advanced (MS, PhD.) degree may substitute for one year of experience	And two years experience in environmental analysis of representative analytes for which they will oversee
Laboratory Director	Bachelor's Degree in science or a minor in chemistry	Or 10 years experience in the environmental laboratory industry
Quality Assurance Manager	Bachelor's degree in a basic or applied science and 1 year nonacademic analytical chemistry; Training in statistics or quality control procedures.	Or 4 years nonacademic analytical chemistry experience.
Technical Director	Bachelor's degree in basic or applied science or engineering and at least 24 college semester credit hours in chemistry	At least two years experience in environmental analysis; master's or doctoral degree may substitute for one year experience.

Analysts-in-training perform tasks under the direct supervision of a qualified analyst or Department supervisor. The person supervising an analyst-in-training is accountable for the quality of the

analytical data. The trainer must review and approve the trainee's data and any associated corrective actions.

18.3 TRAINING

The laboratory is committed to furthering the professional and technical development of employees at all levels.

Orientation to the laboratory's policies and procedures, in-house method training, and employee attendance at outside training courses and conferences all contribute toward employee proficiency. Below are examples of various areas of required employee training:

Required Training	Time Frame*	Employee Type
Environmental Health & Safety	Prior to lab work	All
Ethics – New Hires	1 week of hire	All
Ethics - Comprehensive	90 days of hire	All
Data Integrity	30 days of hire	Technical and PMs
Quality Assurance	90 days of hire	All
Ethics – Comprehensive Refresher	Annually	All
Initial Demonstration of Capability (DOC)	Prior to unsupervised method performance	Technical

The laboratory maintains records of relevant authorization/competence, education, professional qualifications, training, skills and experience of technical personnel (including contracted personnel) as well as the date that approval/authorization was given. These records are kept on file at the laboratory. Also refer to "Demonstration of Capability" in Section 20.

The training of technical staff is kept up to date by:

- Documentation in their training file that they have read, understood and agreed to follow the most recent version of the laboratory QA Manual and SOPs in their area of responsibility. This documentation is updated as SOPs are updated.
- Documentation from any training courses or workshops on specific equipment, analytical techniques or other relevant topics is maintained in their training file.
- Documentation of proficiency (refer to Section 20).
- An Ethics Agreement signed by each staff member (renewed each year) and evidence of annual ethics training.
- A Confidentiality Agreement signed by each staff member signed at the time of employment.
- Human Resources maintains documentation and attestation forms on employment status and records; benefit programs; timekeeping/payroll; and employee conduct (e. g., ethics). This information is maintained in the employee's secured personnel file.
- For each new method for which an analyst demonstrates capability, Figure 18-1, or an equivalent, may be used to summarize that all components of the training have been completed and approved.

Further details of the laboratory's training program are described in the SOP Training / NV08-199.

18.4 DATA INTEGRITY AND ETHICS TRAINING PROGRAM

Establishing and maintaining a high ethical standard is an important element of a Quality System. Ethics and data integrity training is integral to the success of TestAmerica and is provided for each employee at TestAmerica. It is a formal part of the initial employee orientation within 1 week of hire followed by technical data integrity training within 30 days, comprehensive training within 90 days, and an annual refresher for all employees. Senior management at each facility performs the ethics training for their staff.

In order to ensure that all personnel understand the importance TestAmerica places on maintaining high ethical standards at all times, TestAmerica has established an Ethics Policy No. CA-L-P-001 and an Ethics Statement. All initial and annual training is documented by signature on the signed Ethics Statement demonstrating that the employee has participated in the training and understands their obligations related to ethical behavior and data integrity.

Violations of this Ethics Policy are not tolerated. Employees who violate this policy are subject to disciplinary actions up to and including termination. Criminal violations may also be referred to the Government for prosecution. In addition, such actions could jeopardize TestAmerica's ability to do work on Government contracts, and for that reason, TestAmerica has a Zero Tolerance approach to such violations.

Employees are trained as to the legal and environmental repercussions that result from data misrepresentation. Key topics covered in the presentation include:

- Organizational mission and its relationship to the critical need for honesty and full disclosure in all analytical reporting.
- Ethics Policy.
- How and when to report ethical/data integrity issues. Confidential reporting.
- Record keeping.
- Discussion regarding data integrity procedures.
- Specific examples of breaches of ethical behavior (e. g., peak shaving, altering data or computer clocks, improper macros, etc., accepting/offering kickbacks, illegal accounting practices, unfair competition/collusion)
- Internal monitoring. investigations and data recalls.
- Consequences for infractions, including potential for immediate termination, debarment, or criminal prosecution.
- Importance of proper written narration / data qualification by the analyst and project manager with respect to those cases where the data may still be usable but are in one sense or another partially deficient.

Additionally, a data integrity hotline (1-800-736-9407) is maintained by TestAmerica and administered by the Corporate Quality Department.

Figure 18-1. **TRAINING SUMMARY**

Trainee: _____ **Date Training Began:**_____

Topic(s) of Training (circle): **Non-Analytical (General)** **Analytical Method** **Instrument**

SOPs Reviewed: _____

SOPs Signed-off in QA Office: Y / N

I. METHOD/PARAMETER _____

___ Detailed Reference Method/SOP
___ Basic Method/Instrument Theory
___ Safety Precautions
___ Waste Handling
___ Instrument
___ Routine Maintenance
___ Interferences
___ Extraction/Preparation

II. QUALITY CONTROL
___ Calibration Curve, Initial Calibration Verification (ICV) and Continuing Calibration Verification (CCV)
___ Precision/Accuracy
___ MDL Study
___ Review of Chain-of-Custody
___ Documentation (sequences, maintenance, logbooks/worksheets, observations, modifications, standards)
___ QC Requirements (MS/MSD, LCS, Dups, Blanks, Surrogates, Internal Stds, Interference Checks, etc.)
___ Miscellaneous QC (Retention Time Window Studies, IDL)
___ Non-Conformance and Corrective Action Documentation

III. DATA HANDLING AND REPORTING
___ Review Equations and Calculations (concentrations, dry/wet weight)
___ Data Entry or Down-Loading
___ Significant Figures
___ Reporting Dilutions

IV. GENERAL TRAINING
Attach sheet to describe what was discussed. General Training Topics might include: Sample Receiving, Waste Disposal, Shipping, Safety.

Results of Start-up QC :

P&A Results Acceptable Y / N . Attach copy of Precision and Accuracy Results (Summary)

Comments (include any additional training requirements):_____

_____

For AIHA methods, the training period must be a minimum of 20 business days.

Trainee: _____ Date: _____

Trainer: _____ Date: _____

Group Manager: _____ Date: _____

QA Manager: _____ Date: _____

SECTION 19

ACCOMMODATIONS AND ENVIRONMENTAL CONDITIONS
(NELAC 5.5.3)

19.1 OVERVIEW

TestAmerica Nashville is a 40,000 ft^2, secure, laboratory facility with controlled access, designed to accommodate an efficient workflow and to provide a safe and comfortable work environment for employees. All visitors sign in and are escorted by laboratory personnel. Access is controlled by various measures.

The laboratory is equipped with structural safety features. Each employee is familiar with the location, use, and capabilities of general and specialized safety features associated with their workplace. The laboratory provides and requires the use of protective equipment, including safety glasses, protective clothing, gloves, etc. OSHA and other regulatory agency guidelines regarding required amounts of bench and fume hood space, lighting, ventilation (temperature and humidity controlled), access, and safety equipment are met or exceeded.

Traffic flow through sample preparation and analysis areas is minimized to reduce the likelihood of contamination. Adequate floor space and bench top area is provided to allow unencumbered sample preparation and analysis space. Sufficient space is also provided for storage of reagents and media, glassware, and portable equipment. Ample space is also provided for refrigerated sample storage before analysis and archival storage of samples after analysis. Laboratory HVAC and deionized water systems are designed to minimize potential trace contaminants.

The laboratory is separated into specific areas for sample receiving, sample preparation, volatile organic sample analysis, non-volatile organic sample analysis, inorganic sample analysis, and administrative functions.

19.2 ENVIRONMENT

Laboratory accommodation, test areas, energy sources, and lighting are adequate to facilitate proper performance of tests. The facility is equipped with heating, ventilation, and air conditioning (HVAC) systems appropriate to the needs of environmental testing performed at this laboratory.

The environment in which these activities are undertaken does not invalidate the results or adversely affect the required accuracy of any measurements.

The laboratory provides for the effective monitoring, control and recording of environmental conditions that may affect the results of environmental tests as required by the relevant specifications, methods, and procedures. Such environmental conditions include humidity, voltage, temperature, and vibration levels in the laboratory.

When any of the method or regulatory required environmental conditions change to a point where they may adversely affect test results, analytical testing is discontinued until the environmental conditions are returned to the required levels.

Environmental conditions of the facility housing the computer network and LIMS are regulated to protect against raw data loss.

19.3 WORK AREAS

There is effective separation between neighboring areas when the activities therein are incompatible with each other. For example, volatile organic chemical handling areas, including sample preparation and waste disposal, are separated from volatile organic chemical analysis areas.

Access to and use of all areas affecting the quality of analytical testing is defined and controlled by secure access to the laboratory building as described below in the Building Security section.

Adequate measures are taken to ensure good housekeeping in the laboratory and to ensure that any contamination does not adversely affect data quality. These measures include regular cleaning to control dirt and dust within the laboratory.

Work areas are available to ensure an unencumbered work area. Work areas include:

- Access and entryways to the laboratory.
- Sample receipt areas.
- Sample storage areas.
- Chemical and waste storage areas.
- Data handling and storage areas.
- Sample processing areas.
- Sample analysis areas.

19.4 FLOOR PLAN

A floor plan can be found in Appendix 3.

19.5 BUILDING SECURITY

Building keys are distributed to employees as necessary. All doors have automatic closures which lock when closed, except the two main entrances which are locked at 5:30 p.m. each business day.

Visitors to the laboratory sign in and out in a visitors' logbook. A visitor is defined as any person who visits the laboratory who is not an employee of TestAmerica Nashville. The visitors' logbook is used to ensure that everyone gets out of the building safely in case of emergency. The visitor is provided with a "Visitor" identification card to wear while in the laboratory. The Environmental, Health and Safety Manual contains requirements for visitors and vendors. There are specific safety forms that must be reviewed and signed.

Visitors (with the exception of company employees) are escorted by laboratory personnel at all times, or the location of the visitor is noted in the visitors' logbook.

SECTION 20

TEST METHODS AND METHOD VALIDATION
(NELAC 5.5.4)

20.1 OVERVIEW

TestAmerica Nashville uses methods that are appropriate to meet our clients' requirements and that are within the scope of the laboratory's capabilities. These include sampling, handling, transport, storage and preparation of samples, and, where appropriate, an estimation of the measurement of uncertainty as well as statistical techniques for analysis of environmental data.

Instructions are available in the laboratory for the operation of equipment as well as for the handling and preparation of samples. All instructions, Standard Operating Procedures (SOPs), reference methods and manuals relevant to the working of the laboratory are readily available to all staff. Deviations from published methods are documented (with justification) in the laboratory's approved SOPs. SOPs are submitted to clients for review at their request. Significant deviations from published methods require client approval and regulatory approval where applicable.

20.2 STANDARD OPERATING PROCEDURES (SOPS)

TestAmerica Nashville maintains SOPs that accurately reflect all phases of the laboratory, such as assessing data integrity, corrective actions, handling customer complaints as well as all analytical methods and sampling procedures. The method SOPs are derived from the most recently promulgated/approved, published methods and are specifically adapted to the laboratory facility. Modifications or clarifications to published methods are clearly noted in the SOPs. All SOPs are controlled in the laboratory (refer to Section 6 on Document Control):

- All SOPs contain a revision number, effective date, and appropriate approval signatures. Controlled copies are available to all staff.
- Procedures for SOP preparation, review, revision and control are incorporated by reference to SOPs: CW-Q-S-002 (Writing a Standard Operating Procedure (SOP)) and SOP Document Control / NV08-152.
- SOPs are reviewed at a minimum of every two years (annually for Drinking Water and DoD SOPs), and where necessary, revised to ensure continuing suitability and compliance with applicable requirements.

20.3 LABORATORY METHODS MANUAL

For each test method, the laboratory has available the published, referenced method as well as the laboratory developed SOP.

Note: If more stringent standards or requirements are included in a mandated test method or regulation than those specified in this manual, the laboratory demonstrates that such requirements are met. If it is not clear which requirements are more stringent, the standard from the method or regulation is to be followed. Any exceptions or deviations from the referenced methods or regulations are noted in the specific analytical SOP.

The laboratory maintains an SOP Index for both technical and non-technical SOPs. Technical SOPs are maintained to describe a specific test method. Non-technical SOPs are maintained to describe functions and processes not related to a specific test method.

20.4 SELECTION OF METHODS

Since numerous methods and analytical techniques are available, continued communication between the client and laboratory is imperative to assure the correct methods are utilized. Once client methodology requirements are established, this and other pertinent information are summarized by the Project Manager. These mechanisms ensure that the proper analytical methods are applied when the samples arrive for log-in. For non-routine analytical services (e. g., special matrices, non-routine compound lists, etc.), the method of choice is selected based on client needs and available technology. The methods selected must be capable of measuring the specific parameter of interest, in the concentration range of interest, and with the required precision and accuracy.

20.4.1 Sources of Methods

Routine analytical services are performed using standard EPA-approved methodology. In some cases, modification of standard approved methods may be necessary to provide accurate analyses of particularly complex matrices. When the use of specific methods for sample analysis is mandated through project or regulatory requirements, only those methods are used.

When clients do not specify the method to be used or methods are not required, the methods used must be clearly validated and documented in an SOP and available to clients and/or the end user of the data.

20.4.1.1 The analytical methods used by the laboratory are those currently accepted and approved by the U. S. EPA and the state or territory from which the samples were collected. Reference methods include:

- *HASL-300 28th Edition, Environmental Measurements Laboratory (EML), 1997.*
- *Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air, US EPA, January 1996.*
- *Guidelines Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act, and Appendix A-C; 40 CFR Part 136, USEPA Office of Water. Revised as of July 1, 1995, Appendix A to Part 136 - Methods for Organic Chemical Analysis of Municipal and Industrial Wastewater (EPA 600 Series)*
- *Methods for Chemical Analysis of Water and Wastes, EPA 600 (4-79-020), 1983.*
- *Methods for the Determination of Inorganic Substances in Environmental Samples, EPA-600/R-93/100, August 1993.*
- *Methods for the Determination of Metals in Environmental Samples, EPA/600/4-91/010, June 1991. Supplement I: EPA-600/R-94/111, May 1994.*
- *Methods for the Determination of Organic Compounds in Drinking Water, EPA-600/4-88-039, December 1988, Revised, July 1991, Supplement I, EPA-600-4-90-020, July 1990, Supplement II, EPA-600/R-92-129, August 1992. Supplement III EPA/600/R-95/131 - August 1995 (EPA 500 Series) (EPA 500 Series methods)*

- *Technical Notes on Drinking Water Methods, EPA-600/R94-173, October 1994*
- *NIOSH Manual of Analytical Methods, 4th ed., August 1994.*
- *Statement of Work for Inorganics & Organics Analysis, SOM and ISM, current versions, USEPA Contract Laboratory Program Multi-media, Multi-concentration*
- *Standard Methods for the Examination of Water and Wastewater, 18th/19th /20th edition /on-line edition; Eaton, A.D. Clesceri, L.S. Greenberg, A.E. Eds; American Water Works Association, Water Pollution Control Federation, American Public Health Association: Washington, D.C.*
- *Test Methods for Evaluating Solid Waste Physical/Chemical Methods (SW846), Third Edition, September 1986; Final Update I, July 1992; Final Update IIA, August 1993; Final Update II, September 1994; Final Update IIB, January 1995; Final Update III, December 1996; Final Update IIIB, June 2005; and Final Update IV, January 2008.*
- *Annual Book of ASTM Standards, American Society for Testing & Materials (ASTM), Philadelphia, PA.*
- *Manual for the Certification of Laboratories Analyzing Drinking Water (EPA 815-R-05-004, January 2005)*
- *Code of Federal Regulations (CFR) 40, Parts 136, 141, 172, 173, 178, 179 and 261.*

The laboratory reviews updated versions to all the aforementioned references for adaptation based upon capabilities, instrumentation, etc., and implements them as appropriate. As such, the laboratory strives to perform the latest versions of each approved method as regulations allow or require.

Other reference procedures for non-routine analyses may include methods established by specific states (e.g., Underground Storage Tank methods), ASTM or equipment manufacturers. Sample type, source, and the governing regulatory agency requiring the analysis determines the method utilized.

The laboratory must inform the client when a method proposed by the client may be inappropriate or out of date. After the client has been informed, and they wish to proceed contrary to the laboratory's recommendation, it must be documented.

20.4.2 Demonstration of Capability

Before the laboratory may institute a new method and begin reporting results, the laboratory must confirm that it can properly operate the method. In general, this demonstration does not test the performance of the method in real world samples, but in an applicable and available clean matrix sample. If the method is for the testing of analytes that are not conducive to spiking, demonstration of capability may be performed on quality control samples.

20.4.2.1 A demonstration of capability is performed whenever there is a change in instrument type, method or personnel. A Demonstration of Capability is also performed when an instrument (new or used) is moved into a department and brought online for the first time in that analytical department.

20.4.2.2 The initial demonstration of capability must be thoroughly documented and approved by the Technical Director and QA Manager prior to independently analyzing client samples. All associated documentation must be retained in accordance with the

laboratory's archiving procedures. Refer to Section 15, Control of Records and SOP Training / NV08-199.

20.4.2.3 The laboratory must have an approved SOP, demonstrate satisfactory performance, and conduct a method detection limit study (when applicable). There may be other requirements as stated within the published method or regulations (i. e., retention time window study). See SOP Method Startup / NV08-203.

Note: In some instances, a situation may arise where a client requests that an unusual analyte be reported using a method where this analyte is not normally reported. If the analyte is being reported for regulatory purposes, the method must meet all procedures outlined within this QA Manual (SOP, MDL, and Demonstration of Capability). If the client states that the information is not for regulatory purposes, the result may be reported as long as the following criteria are met:

- The instrument is calibrated for the analyte to be reported using the criteria for the method and ICV/CCV criteria are met (unless an ICV/CCV is not required by the method or criteria are per project specifications).
- The laboratory's nominal or default reporting limit (RL) is equal to the quantitation limit (QL), must be at or above the lowest non-zero standard in the calibration curve and must be reliably determined. Project RLs (determined during Work Request Review, see Section 7) are client specified reporting limits which may be higher than the QL. Results reported below the QL must be qualified as estimated values. Also see Section 20.6.1.3, Relationship of Limit of Detection (LOD) to Quantitation Limit (QL).

  Note: For the purposes of Ohio VAP, the term reporting limit (RL) is used exclusively by the laboratory.

- The client request is documented and the lab informs the client of its procedure for working with unusual compounds. The final report must be footnoted: *Reporting Limit based on the low standard of the calibration curve.*
- Refer to Section 12 (Control of Non-Conforming Work).

20.4.3 Initial Demonstration of Capability (IDOC) Procedures

20.4.3.1 The spiking standard used must be prepared independently from those used in instrument calibration.

20.4.3.2 The analyte(s) are diluted in a volume of clean matrix sufficient to prepare four aliquots at the concentration specified by a method or the laboratory SOP or if unspecified to a concentration 1-4 times the laboratory RL.

20.4.3.3 At least four aliquots are prepared (including any applicable clean-up procedures) and analyzed according to the test method (either concurrently or over a period of days). Note: Results of successive LCS analyses can be used to fulfill the DOC requirement.

20.4.3.4 Using all of the results, calculate the mean recovery in the appropriate reporting units and the standard deviations for each parameter of interest.

20.4.3.5 When it is not possible to determine the mean and standard deviations, such as for presence, absence and logarithmic values, the laboratory assesses performance against criteria described in the Method SOP.

20.4.3.6 Compare the information obtained above to the corresponding acceptance criteria for precision and accuracy in the test method (if applicable) or in laboratory generated acceptance criteria (LCS or interim criteria) if there is no mandatory criteria established. If any one of the parameters do not meet the acceptance criteria, the performance is unacceptable for that parameter.

20.4.3.7 When one or more of the tested parameters fail at least one of the acceptance criteria, the analyst must proceed according to either option listed below:

- Locate and correct the source of the problem and repeat the test for all parameters of interest beginning with 20.4.3.3 above.
- Beginning with 20.4.3.3 above, repeat the test for all parameters that failed to meet criteria. Repeated failure, however, confirms a general problem with the measurement system. If this occurs, locate and correct the source of the problem and repeat the test for all compounds of interest beginning with 20.4.3.1 above.

A certification statement (see Figure 20-1) is used to document the completion of each initial demonstration of capability. A copy of the certification is archived in the analyst's training folder.

Methods on line prior to the effective date of this Section must be updated to the procedures outlined above as new analysts perform their demonstration of capability. A copy of the new record replaces that which was used for documentation in the past. At a minimum, the precision and accuracy of four mid-level laboratory control samples must have been compared to the laboratory's quality control acceptance limits.

20.5 LABORATORY DEVELOPED METHODS AND NON-STANDARD METHODS

Any new method developed by the laboratory must be fully defined in an SOP and validated by qualified personnel with adequate resources to perform the method. Method specifications and the relation to client requirements must be clearly conveyed to the client if the method is a non-standard method (not a published or routinely accepted method). The client must also be in agreement to the use of the non-standard method.

20.6 VALIDATION OF METHODS

Validation is the confirmation by examination and the provision of objective evidence that the particular requirements for a specific intended use are fulfilled.

All non-standard methods, laboratory designed/developed methods, standard methods used outside of their scope, and major modifications to published methods must be validated to confirm they are fit for their intended use. The validation is as extensive as necessary to meet

the needs of the given application. The results are documented with the validation procedure used and contain a statement as to the fitness for use. (See SOP Method Startup / NV08-203.)

20.6.1 Method Validation and Verification Activities for All New Methods

While method validation can take various courses, the following activities can be required as part of method validation. Method validation records are designated QC records and are archived accordingly.

20.6.1.1 Determination of Method Selectivity

Method selectivity is the demonstrated ability to discriminate the analyte(s) of interest from other compounds in the specific matrix or matrices from other analytes or interference. In some cases to achieve the required selectivity for an analyte, a confirmation analysis is required as part of the method.

20.6.1.2 Determination of Method Sensitivity

Sensitivity can be both estimated and demonstrated. Whether a study is required to estimate sensitivity depends on the level of method development required when applying a particular measurement system to a specific set of samples. Where estimations and/or demonstrations of sensitivity are required by regulation or client agreement, such as the procedure in 40 CFR Part 136 Appendix B, under the Clean Water Act, these must be followed. The laboratory determinations of MDLs are described in Section 20.7.

20.6.1.3 Relationship of Limit of Detection (LOD) to the Quantitation Limit (QL)

An important characteristic of expression of sensitivity is the difference in the LOD and the QL. The LOD is the minimum level at which the presence of an analyte can be reliably detected. The QL is the minimum concentration of analyte that can be quantitatively determined with acceptable precision and bias. For most instrumental measurement systems, there is a region where semi-quantitative data is generated around the LOD (both above and below the estimated MDL or LOD) and below the QL. In this region, detection of an analyte may be confirmed but quantification of the analyte is unreliable within the accuracy and precision guidelines of the measurement system. When an analyte is detected below the QL, and the presence of the analyte is confirmed by meeting the qualitative identification criteria for the analyte, the analyte can be reliably reported, but the amount of the analyte can only be estimated. If data is to be reported in this region, it must be done so with a qualification that denotes the semi-quantitative nature of the result.

Note: For the purposes of Ohio VAP, the terms MDL and reporting limit (RL) are used exclusively by the laboratory.

20.6.1.4 Determination of Interferences

A determination that the method is free from interferences in a blank matrix is performed.

20.6.1.5 Determination of Range

Where appropriate to the method, the quantitation range is determined by comparison of the response of an analyte in a curve to established or targeted criteria. Generally the upper quantitation limit is defined by highest acceptable calibration concentration. The lower quantitation limit or QL cannot be lower than the lowest non-zero calibration level, and can be constrained by required levels of bias and precision.

20.6.1.6 Determination of Accuracy and Precision

Accuracy and precision studies are generally performed using replicate analyses, with a resulting percent recovery and measure of reproducibility (standard deviation, relative standard deviation) calculated and measured against a set of target criteria.

20.6.1.7 Documentation of Method

The method is formally documented in an SOP. If the method is a minor modification of a standard laboratory method that is already documented in an SOP, an SOP Attachment describing the specific differences in the new method is acceptable in place of a separate SOP.

20.6.1.8 Continued Demonstration of Method Performance

Continued demonstration of Method Performance is addressed in the SOP. Continued demonstration of method performance is generally accomplished by batch specific QC samples such as LCS, method blanks or PT samples.

20.7 METHOD DETECTION LIMITS (MDL) / LIMITS OF DETECTION (LOD)

Method detection limits (MDL) are initially determined in accordance with 40 CFR Part 136, Appendix B or alternatively by other technically acceptable practices that have been accepted by regulators. MDL is also sometimes referred to as Limit of Detection (LOD). The MDL theoretically represents the concentration level for each analyte within a method at which the Analyst is 99% confident that the true value is not zero. The MDL is determined for each analyte initially during the method validation process and updated as required in the analytical methods, whenever there is a significant change in the procedure or equipment, or based on project specific requirements (refer to 20.7.10). The analyst prepares at least seven replicates of solution spiked at one to five times the estimated method detection limit (most often at the lowest standard in the calibration curve) into the applicable matrix with all the analytes of interest. Each of these aliquots is extracted (including any applicable clean-up procedures) and analyzed in the same manner as the samples. Where possible, the seven replicates are analyzed over 2-4 days to provide a more realistic MDL.

Note: For the purposes of Ohio VAP, the term MDL is used exclusively by the laboratory.

20.7.1 MDLs are initially performed for each individual instrument and non-microbiological method analysis. Unless there are requirements to the contrary, the laboratory uses the highest calculated MDL for all instruments used for a given method as the MDL for reporting purposes. This MDL is not required for methods that are not readily spiked (e. g., pH, turbidity, etc.) or

where the lab does not report values to the MDL. For titration and gravimetric methods where there is no additional preparation involved, the MDL is based on the lowest discernable unit of measure that can be observed.

20.7.2 MDLs must be run against acceptable instrument QC, including ICVs and Tunes. This is to insure that the instrument is in proper working condition and falsely high or low MDLs are not calculated.

20.7.3 Use only clean matrix which is free of target analytes (e. g., Laboratory reagent water, Ottawa Sand) unless a project specific MDL is required in a field sample matrix.

20.7.4 For Ohio VAP, the MDL spike concentration must not exceed the reporting limit.

20.7.5 The Reporting Limit (also may be referred to as Limit of Quantitation or LOQ) is generally between 2 and 5 times the MDL. If the MDL is being performed during method development, use this guideline to determine the Reporting Limit for the analysis. For DoD, the RL is $\geq$ 3x MDL. If a sample is diluted or nominal prep volume.weight is vaired, the reported MDL and RL are adjusted according to the dilution factor.

20.7.6 There are three criteria for success:

- 10 times the MDL must be the spike concentration; if not, the MDL is set at 1/3 the RL. (The RL cannot be less than the spike concentration.)
- Recovery must be between 50 and 150% of the true value (unless specified otherwise in the method SOP, and
- The ratio of the RL to the MDL should be greater than two (preferably three of more).

If the MDL must be repeated to achieve these criteria, extraction or digestion must also be repeated. A higher spike level may be necessary. The test concentration used for the MDL calculation becomes the new RL.

20.7.7 The calculated MDL cannot be greater than the spike amount. If this occurs, repeat or increase the spike amount.

20.7.8 If the most recently calculated MDL does not permit qualitative identification of the analyte, then the laboratory may use technical judgment for establishing the MDL (e. g., calculate what level would give a qualitative ID, compare with IDL (Section 20.8), spike at a level where qualitative ID is determined and assign that value as MDL, minimum sensitivity requirements, Standard deviation of method blanks over time, etc.). These alternate verification procedures must be documented in the laboratory SOP on MDLs (MDL / NV08-202).

20.7.9 Each of the seven spikes must be qualitatively identifiable (e. g., appear in both columns for dual column methods, characteristic ions for GCMS mass spectra, etc.). Manual integrations to force the baseline for detection are not allowed.

20.7.10 The initial MDL is calculated as follows:

$$MDL = t_{(n-1,\ 1-a\ =\ 0.99)} \times (\text{Standard Deviation of replicates})$$

where $t_{(n-1,\ 1-a\ =\ 0.99)} = 3.143$ for seven replicates.

20.7.11 Subsequent to the initial MDL determination, periodic MDL verification, confirmation or determinations may be performed by the procedure in 40 CFR Part 136, Appendix B or alternatively by other technically acceptable practices (e. g., method blanks over time, single standard spikes that have been subjected to applicable sample prep processes, etc.).

20.7.12 Because of the inherent variability in results outside of the calibration range, TestAmerica does not recommend the reporting of results below the lowest calibration point in a curve; however, it is recognized that some projects and agencies require the reporting of results below the RL. Any result that falls between the MDL and the Reporting limit, when reported, must be qualified as an estimated value.

20.7.13 Detections reported down to the MDL must be qualitatively identified.

20.7.14 MDLs and reporting limits are adjusted in LIMs based on moisture content and sample aliquot size.

20.7.15 Refer to the Corporate SOP No. CA-Q-S-006 or the laboratory's SOP MDL/NV08-202 for details on the laboratory's MDL process.

20.8 INSTRUMENT DETECTION LIMITS (IDL)

20.8.1 The IDL is sometimes used to assess the reasonableness of the MDLs or in some cases required by the analytical method or program requirements. IDLs are most used in metals analyses but may be useful in demonstration of instrument performance in other areas.

20.8.2 IDLs are calculated to determine an instrument's sensitivity independent of any preparation method. IDLs are calculated either using seven replicate spike analyses, like MDL but without sample preparation, or by the analysis of 10 instrument blanks and calculating 3 x the absolute value of the standard deviation.

20.8.3 If IDL is > than the MDL, it may be used as the reported MDL.

20.9 VERIFICATION OF DETECTION AND REPORTING LIMITS

20.9.1 Once an MDL is established, it must be verified, on each instrument, by analyzing a quality control sample (prepared as a sample) at approximately 2-3 times the calculated MDL for single analyte analyses (e. g., most wet chemistry methods, etc.) and 1-4 times the calculated MDL for multiple analyte methods (e. g., GC, GCMS, ICP, etc.). The analytes must be qualitatively identified or see Sections 20.7.8 and 20.7.11 for other options. This verification does not apply to methods that are not readily spiked (e. g., pH, turbidity, etc.) or where the lab does not report to the MDL. If the MDL does not verify, then the lab must not report to the MDL, or redevelop their MDL or use the level where qualitative identification is established (see 20.7). MDLs must be verified at least annually.

20.9.2 When the laboratory establishes a quantitation limit, it must be initially verified by the analysis of a low level standard or QC sample at 1-2 the reporting limit and annually thereafter. The annual requirement is waved for methods that have an annually verified MDL. The laboratory must comply with any regulatory requirements.

20.10 RETENTION TIME WINDOWS

Most organic analyses and some inorganic analyses use chromatography techniques for qualitative and quantitative determinations. For every chromatography analysis, each analyte has a specific time of elution from injection on column to the detector. This is known as the analyte's retention time. The variance in the expected time of elution is defined as the retention time window. As the key to analyte identification in chromatography, retention time windows must be established on every column for every analyte used for that method. These records are kept with the files associated with an instrument for later quantitation of the analytes.

For GC, HPLC and IC methods, there must be sufficient separation between analyte peaks so as to not misidentify analytes. In the mid-level standard, the distance between the valley and peak height cannot be any less than 25% of the sum of the peak heights of the analytes. This also applies to GCMS in the case where the two compounds share the same quantitation ion.

Note: Some analytes do not separate sufficiently to be able to identify or quantitate them as separate analytes (e. g., m-xylene and p-xylene) and are quantitated and reported as a single analyte (e. g., m, p-xylenes).

Once the analyst has determined that the instrument is in optimum working condition through calibration and calibration verification procedures, he or she uses a mid-range calibration or calibration verification standard to establish the retention times for each of the individual analytes in a method. The analyst makes three injections of the same standard over a 72-hour (24 hr-period for 300.0) period, tabulating the retention times for each analyte for each of the three injections. The width of retention time window is normally the average absolute retention time ± 3 Standard Deviations. A peak outside the retention time window is not identified by the computer as a positive match of the analyte of interest.

Record the retention time for each single component analyte and surrogate to three decimal places (e. g., 0.007). Calculate the mean and standard deviation of the three absolute retention times for each single component analyte and surrogate. For multi-component analytes, choose three to five major peaks (see the determinative methods for more details) and calculate the mean and standard deviation of those peaks.

If the standard deviation of the retention times for a target compound is between 0.000 and 0.010, then the laboratory may either collect data from additional injections of standards or use a default standard deviation of 0.01 minutes.

The width of the retention time window for each analyte, surrogate, and major constituent in multi-component analytes is defined as ± 3 times the standard deviation of the mean absolute retention time established during the 72 hour period. If the default standard deviation in (c) is employed, the width of the window is set at 0.03 minutes.

Establish the center of the retention time window for each analyte and surrogate by using the absolute retention time for each analyte and surrogate from the calibration verification standard at the beginning of the analytical shift. For samples run during the same shift as an initial calibration, use the retention time of the mid-point standard of the initial calibration.

The laboratory must calculate absolute retention time windows for each analyte and surrogate on each chromatographic column and instrument, except GC/MS.

If the instrument data system is not capable of employing compound-specific retention time windows, then the analyst may choose a window that minimizes false negatives and positives and apply it to all compounds. As noted above, other approaches may also be employed, but must be documented by the analyst. In general, you do not use a window greater than 0.2 to 0.3 minutes. If windows larger than this have been determined, a cause must be looked for and the windows should be re-determined.

It is possible for the statistically calculated RT window to be too tight and need to be adjusted based on analyst experience. In these instances method default retention time windows may be used (e. g., for 8000 series methods a default of 0.03 minutes may be used, and EPA CLP 0.05 minutes is used). The same concept is applied when any peak outside of that window is not identified by the computer as a positive match.

The calibration verification standard at the beginning of a run may be used to adjust the RT for an analyte. This is essentially re-centering the window but the size of the window remains the same. The RTs are verified when all analytes are within their RT windows and are properly identified.

20.11 EVALUATION OF SELECTIVITY

The laboratory evaluates selectivity by following the checks within the applicable analytical methods, which include mass spectral tuning, second column confirmation, ICP interelement interference checks, chromatography retention time windows, sample blanks, spectrochemical, atomic absorption or fluorescence profiles, co-precipitation evaluations and specific electrode response factors.

20.12 ESTIMATION OF UNCERTAINTY OF MEASUREMENT

20.12.1 Uncertainty is "a parameter associated with the result of a measurement, that characterizes the dispersion of the values that could reasonably be attributed to the measurand" (as defined by the International Vocabulary of Basic and General Terms in Metrology, ISO Geneva, 1993, ISBN 92-67-10175-1). Knowledge of the uncertainty of a measurement provides additional confidence in a result's validity. Its value accounts for all the factors which could possibly affect the result, such as adequacy of analyte definition, sampling, matrix effects and interferences, climatic conditions, variances in weights, volumes, and standards, analytical procedure, and random variation. Some national accreditation organizations require the use of an "expanded uncertainty": the range within which the value of the measurand is believed to lie within at least a 95% confidence level with the coverage factor k=2.

20.12.2 Uncertainty is not error. Error is a single value, the difference between the true result and the measured result. On environmental samples, the true result is never known. The measurement is the sum of the unknown true value and the unknown error. Unknown error is a combination of systematic error, or bias, and random error. Bias varies predictably, constantly, and independently from the number of measurements. Random error is unpredictable, assumed to be Gaussian in distribution, and reducible by increasing the number of measurements.

20.12.3 The minimum uncertainty associated with results generated by the laboratory can be determined by using the Laboratory Control Sample (LCS) accuracy range for a given analyte. The LCS limits are used to assess the performance of the measurement system since they take

into consideration all of the laboratory variables associated with a given test over time (except for variability associated with the sampling and the variability due to matrix effects). The percent recovery of the LCS is compared either to the method-required LCS accuracy limits or to the statistical, historical, in-house LCS accuracy limits.

20.12.4 To calculate the uncertainty for the specific result reported, multiply the result by the decimal of the lower end of the LCS range percent value for the lower end of the uncertainty range, and multiply the result by the decimal of the upper end of the LCS range percent value for the upper end of the uncertainty range. These calculated values represent a 99%-certain range for the reported result. As an example, suppose that the result reported is 1.0 mg/l, and the LCS percent recovery range is 50 to 150%. The uncertainty range would be 0.5 to 1.5 mg/l, which could also be written as 1.0 +/- 0.5 mg/l.

20.12.5 In the case where a well recognized test method specifies limits to the values of major sources of uncertainty of measurement (e. g., 524.2, 525, etc) and specifies the form of presentation of calculated results, no further discussion of uncertainty is required. Note: This section does not apply to AIHA.

20.13 CONTROL OF DATA

The laboratory has policies and procedures in place to ensure the authenticity, integrity, and accuracy of the analytical data generated by the laboratory.

20.13.1 Computer and Electronic Data Related Requirements

The three basic objectives of our computer security procedures and policies are shown below. More detail is outlined in SOP LIMS / NV09-12 and Electronic Lab Data Archiving / NV09-171. The laboratory is currently running Element Datasystem which is a third party LIMS system that has been highly customized to meet the needs of the laboratory. It is referred to as LIMS for the remainder of this section. The LIMS utilizes Microsoft SQL Server 2005 which is an industry standard relational database platform. It is referred to as Database for the remainder of this section.

20.13.1.1 Maintain the Database Integrity: Assurance that data is reliable and accurate through data verification (review) procedures, password-protecting access, anti-virus protection, data change requirements, as well as an internal LIMS permissions procedure.

- LIMS Database Integrity is achieved through data input validation, internal user controls, and data change requirements.
- Spreadsheets and other software developed in-house must be verified with documentation through hand calculations prior to use.

  Note: "Commercial off-the-shelf software in use within the designed application range is considered to be sufficiently validated." *From NELAC 2003 Standard.* However, laboratory specific configurations or modifications are validated prior to use.

- In order to assure accuracy, all data entered or transferred into the LIMS data system goes through a minimum of two levels of review.

- The QA department performs random data audits to ensure the correct information has been reported.
- Analytical data file security is provided through three policies.
  - The first policy forbids unauthorized personnel from using laboratory data acquisition computers.
  - The second policy is the implementation of network passwords and login names that restrict directory access.
  - The third layer is maintained through the LIMS and includes the use of username/password combinations to gain access to the LIMS system, the fact that all data in the LIMS is associated with the user to added/reviewed the data, and the restriction of review authority of data.
- All software installations must be in accordance with any relevant copyright licensing regulations.
- All software installed on any computer within the laboratory must be approved by the Information Technology department regional support technician assigned to the laboratory. Shrink-wrapped or otherwise sealed OEM software that is directly related to instrument usage does not need approval, but the Information Technology department must be notified of the installation.
- Anti-virus software is installed on all servers and workstations. The anti-virus software is configured to check for virus signature file and program updates on a daily basis, and these updates are pushed to all servers and workstations. The anti-virus software is configured to clean any virus-infected file, if possible; otherwise, the file is deleted. Disks and CDs brought from any outside source that are not OEM software must be scanned for viruses before being accessed.
- The clocks on data acquisition systems for instruments may only be changed by IT, a department supervisor, a manager, or a director, and the change must be documented in the maintenance log. The Element clock can only be changed by the IT department.

- **Interlab LIMS Permissions Policy**
  - PURPOSE - The purpose of this policy is to provide a mechanism for maintaining the integrity of information contained in each laboratory's LIMS while providing the necessary access for information sharing to staff at other laboratory facilities.
  - DEFINITIONS - Host Laboratory: The laboratory facility that 'owns' the LIMS system or 'hosts' a project/job.
  - POLICIES

    (a) All permissions for the laboratory's LIMS system must only be granted by a representative of that laboratory.
    - If someone outside of the host lab needs permissions for Project Management or other uses, they must go through the Lab Director or his/her designated representative.
    - Permissions must never be granted without the knowledge of the host laboratory.

    (b) Only laboratory analytical, Senior PMs, or QA staff from the home laboratory may have edit permissions for laboratory analysis data.

    (c) Any changes made in laboratory's LIMS system:
    - Must be documented and traceable.
    - If made by staff of an affiliate lab, written permission from the home lab to make the changes (email approval is sufficient) is required.

- No corrections may be made in another laboratory's system without their knowledge.

(d) Data qualifiers in laboratory reports must only be corrected, edited, etc. by the staff at the host laboratory.
(e) Full analytical data "View" only permissions may be granted to outside Project Management and Sales staff. Search permissions may also be granted so status may be checked.
(f) All qualifiers must be approved by the lab QA Manager before adding to standard reference tables (Static tables).
(g) Please contact Corporate QA or IT staff if you have any questions regarding implementation or interpretation of the Interlab LIMS Permissions policy.

20.13.1.2 **Ensure Information Availability:** Protection against loss of information or service is ensured through scheduled back-ups, stable file server network architecture, secure storage of media, line filter, Uninterruptible Power Supply (UPS), and maintaining older versions of software as revisions are implemented.

- Insured by timely backup procedures on reliable backup media, stable file server network architecture, and UPS protection
- UPS Protection:
  - Each fileserver is protected by an appropriate power protection/backup unit. In the event of a power outage, there is approximately 15-30 minutes of up-time for the servers prior to shutdown. This allows for proper shutdown procedures to be followed with the fileservers.
- File Server Architecture
  - All files are maintained on multiple Windows 2000 or newer servers which are secured physically in the Information Technology office. Access to these servers is limited to members of the Information Technology staff.
  - All supporting software is maintained for at least 10 years from the last raw data generated using that software. (Length of time is dependent on local regulations or client requirements (e. g., OH VAP requires 10 years).)
- System Back-up Overview and Procedures
  - Data from both servers and instrument attached PCs are backed up and purged in compliance with the corporate back-up policy.
  - A Maintenance Plan has been defined to create a daily archive of all data within the LIMS database to a backup location. This backup is initiated automatically by either the database or back-up system.
  - Backup tapes are stored in compliance with the corporate Data Backup Policy. Backup verifications are carried out in accordance with the corporate Data Backup Policy.
  - Instrument data back-ups are verified on a periodic basis by the QA department when performing electronic data audits. The audit takes place on data that has been moved to a back-up location ensuring that it has been moved.

20.13.1.3 Maintain Confidentiality: Ensure data confidentiality through physical access controls of when electronically transmitting data.

- All servers are located in a secure area of the IT department offices. Access to the servers is limited to IT staff members and senior lab management.

- The company website contains SSL (Secure Socket Layer) encryption for secure website sessions and data transfers.
- The reporting portion of the LIMS system requires a project manager to enter their unique password anytime they create a report that displays a signature on it (.PDF).
- Electronic documents such as PDF files and electronic data deliverables are made available to clients via the secure web site. The logon page for this web site contains an agreement which states that the customer agrees not to alter any electronic data made available to them. The customer must accept this agreement before they are granted access the web site.

20.13.2 Data Reduction

The complexity of the data reduction depends on the analytical method and the number of discrete operations involved (e. g., extractions, dilutions, instrument readings and concentrations). The analyst calculates the final results from the raw data or uses appropriate computer programs to assist in the calculation of final reportable values.

Manual integration of peaks must be documented and reviewed, and the raw data must be flagged in accordance with Section 20.13.5 and the TestAmerica Corporate SOP CA-Q-S-002, *Acceptable Manual Integration Practices*.

Analytical results are reduced to appropriate concentration units specified by the analytical method, taking into account factors such as dilution, sample weight or volume, etc. Blank correction must be applied only when required by the method or per manufacturer's indication; otherwise, it is not performed. Calculations are independently verified by appropriate laboratory staff.

20.13.2.1 All raw data must be retained in the worklist folder, computer file (if appropriate), and/or runlog. All criteria pertinent to the method must be recorded. The documentation is recorded at the time observations or calculations are made and must be signed or initialed/dated (month/day/year). It must be easily identifiable who performed which tasks if multiple people were involved.

20.13.2.2 In general, concentration results are reported in milligrams per liter (mg/L) or micrograms per liter (µg/L) for liquids and milligrams per kilogram (mg/kg) or micrograms per kilogram (µg/kg) for solids. The units "mg/L" and "mg/kg" are the same as "parts per million (ppm)". The units "µg/L" and "µg/kg" are the same as "parts per billion (ppb)." For values greater than 10,000 mg/L, results can be reported in percent, i. e., 10,000 mg/L = 1%.

- Several environmental methods, such as color, turbidity, conductivity, use very specific, non-concentration units to report results (e. g., NTU, umhos/cm etc).
- Occasionally, the client requests that results be reported in units which take into account the measured flow of water or air during the collection of the sample. When they provide this information, the calculations can be performed and reported.

20.13.2.3 The rounding rule is: round up if the digit to be discarded is larger than 5; round down if the digit to be discarded is less than 5. If the digit is exactly 5, round down if the preceding digit is even; round up if the preceding digit is odd.

20.13.2.4 In reporting, the analyst or the instrument output records the raw data result using values of known certainty plus one uncertain digit. If final calculations are performed external to LIMS, the results should be reported to three significant figures. In general, results are reported to three significant figures on the final report.

20.13.2.5 For those methods that do not have an instrument printout or an instrumental output compatible with the LIMS System, the raw results and dilution factors are entered directly into LIMS by the analyst, and the software calculates the final result for the analytical report. LIMS has a defined significant figure criterion for each analyte. Manually entered results are typed into LIMS in mg/L or mg/kg, except for low-level phenolics (ug/L) and parameters without mass-specific concentration measuring units (such as, NTU, micromhos, etc.).

20.13.2.6 The laboratory strives to import data directly from instruments or calculation spreadsheets to ensure that the reported data are free from transcription and calculation errors. For those analyses with an instrumental output compatible with the LIMS, the raw results and dilution factors are transferred into LIMS electronically after reviewing the quantitation report, and removing unrequested or poor spectrally-matched compounds. The analyst then checks the data in Element against the raw data, and the instrument printout of calibrations, concentrations, retention times, chromatograms, and mass spectra, if applicable, are retained with the data file. The data file is stored in a monthly folder on the instrument computer; periodically, this file is transferred to the server and, eventually, to a tape file.

20.13.3 Logbook / Worksheet Use Guidelines

Logbooks and worksheets are filled out "real time" and have enough information on them to trace the events of the applicable analysis/task (e. g., calibrations, standards, analyst, sample ID, date, time on short holding time tests, temperatures when applicable, calculations are traceable, etc.)

- The following information is required to be documented in Analytical Run Logs:
    - Method reference
    - Instrument ID
    - Sequence of standards, blanks, and samples including: dilutions, explanation of anomalies, standard ID numbers
    - Date of analytical batch
    - Analyst initials and date (Note: Initials across the pages if sheet taped in)
    - QC recoveries and RPDs (required for single target analyte methods only)
    - Any empty space crossed out with a "Z", dated and initialed
    - Reference to ID numbers of standards used
    - Evidence of department supervisor review on a monthly basis
    - pH/residual chlorine checks (volatiles and volatiles UST)
    - Reference to Maintenance log (when maintenance required)

- The following information is required to be documented in Prep Logs and/or Prep Sheets:
    - Method reference
    - Balance ID
    - Sequence of standards, blanks, and samples including: dilutions, explanation of anomalies, standard ID numbers, solvent ID numbers
    - Date and time of preparation
    - Analyst initials and date
    - How QC is prepared
    - Any empty space is crossed out with a "Z", dated and initialed.
    - Evidence of department supervisor review on a monthly basis
    - pH/residual chlorine checks

- Corrections are made following the procedures outlined in Section 13.
- Logbooks are controlled by the QA department. A record is maintained of all logbooks in the lab.
- Unused portions of pages must be "Z"'d out, signed and dated.
- Worksheets are created with the approval of the Technical Director or QA Manager at the facility. The QA Manager controls all worksheets following the procedures in Section 6.

20.13.4 Review / Verification Procedures

Review procedures are outlined in several SOPs (e. g., Sample Control / NV02-01, Project Management / SA02-174, Data Package Procedure / NV08-139) to ensure that reported data are free from calculation and transcription errors, that QC parameters have been reviewed and evaluated before data is reported. The laboratory also has an SOP discussing Manual Integrations to ensure the authenticity of the data (CA-Q-S-002). The general review concepts are discussed below, more specific information can be found in the SOPs.

20.13.4.1 The data review process at TestAmerica Nashville starts at the Sample Control level. Sample Control personnel review chain-of-custody forms and input the sample information and required analyses into a computer LIMS. The Sample Control Supervisor reviews the transaction of the chain-of-custody forms and the inputted information. The Project Managers perform final review of the chain-of-custody forms and inputted information.

20.13.4.2 The next level of data review occurs with the analysts. As results are generated, analysts review their work to ensure that the results generated meet QC requirements and relevant EPA methodologies. The analysts transfer the data into the LIMS and add data qualifiers if applicable (see Appendix 7 for list of common data qualifiers). To ensure data compliance, a department supervisor or group leader (or other designee) performs a second level of review. Second level review is accomplished by checking

reported results against raw data and evaluating the results for accuracy. During the second level review, blank runs, QA/QC check results, initial and continuing calibration results, laboratory control samples, sample data, qualifiers and spike information are evaluated. Where calibration is not required on a daily basis, secondary review of the initial calibration results may be conducted at the time of calibration. Data review checklists are used to document both analyst and second level reviews. Issues that deem further review include the following:

- QC data are outside the specified control limits for accuracy and precision
- Reviewed sample data does not match with reported results
- Unusual detection limit changes are observed
- Raw data indicating some type of contamination or poor technique
- Inconsistent peak integration
- Transcription errors
- Results outside of calibration range

20.13.4.3 Unacceptable analytical results may require reanalysis of the samples. Corrective action for analytical non-conformances is documented in the PIPE database. Problems may also be brought to the attention of the Laboratory Director, Project Manager, Quality Assurance Manager, Technical Director, Operations Manager, or Department Supervisor for further investigation.

20.13.4.4 As a final review prior to the release of the report, the Project Manager reviews the results for appropriateness and completeness. This review and approval ensures that client requirements have been met and that the final report has been properly completed. The process includes, but is not limited to, verifying that chemical relationships are evaluated, COC is followed, cover letters/ narratives are present, flags are appropriate, and project specific requirements are met. The following are some examples of chemical relationships that are reviewed (if data is available):

- Total Results are $\geq$ Dissolved results (e.g. metals)
- Total Solids (TS) $\geq$ TDS or TSS
- TKN $\geq$ Ammonia
- Total Phosphorus $\geq$ Orthophosphate
- COD $\geq$ TOC
- Total cyanide $\geq$ Amenable Cyanide
- TDS $\geq$ individual anions
- Total Chromium $\geq$ Hexavalent Chromium
- BOD $\geq$ cBOD

20.13.4.5 Any project that requires a data package is subject to a tertiary data review for transcription errors and acceptable quality control requirements. The Project

Manager then signs the final report. (*Also see section 26 on Reporting Results*). The accounting personnel also check the report for any clerical or invoicing errors. When complete, the report is sent out to the client.

20.13.4.6 A visual summary of the flow of samples and information through the laboratory, as well as data review and validation, is presented in Figure 20-3.

20.13.5 Manual Integrations

Computerized data systems provide the analyst with the ability to re-integrate raw instrument data in order to optimize the interpretation of the data. Though manual integration of data is an invaluable tool for resolving variations in instrument performance and some sample matrix problems, when used improperly, this technique would make unacceptable data appear to meet quality control acceptance limits. Improper re-integrations lead to legally indefensible data, a poor reputation, or possible laboratory decertification. Because guidelines for re-integration of data are not provided in the methods and most methods were written prior to widespread implementation of computerized data systems, the laboratory trains all analytical staff on proper manual integration techniques using SOP CA-Q-S-002 as the guidelines.

20.13.5.1 The analyst must adjust baseline or the area of a peak in some situations, for example when two compounds are not adequately resolved or when a peak shoulder needs to be separated from the peak of interest. The analyst must use professional judgment and common sense to determine when manual integrating is required. Analysts are encouraged to ask for assistance from a senior analyst or manager when in doubt.

20.13.5.2 Analysts must not increase or decrease peak areas for the sole purpose of achieving acceptable QC recoveries that would have otherwise been unacceptable. The intentional recording or reporting of incorrect information (or the intentional omission of correct information) is against company principals and policy and is grounds for immediate termination.

20.13.5.3 Client samples, performance evaluation samples, and quality control samples are all treated equally when determining whether or not a peak area or baseline should be manually adjusted.

20.13.5.4 All manual integrations are documented by printing before and after chromatograms/spectra with a brief explanation of why the integration was performed. The chromatograms/spectra must be scaled in such a way that the reviewer can easily identify the changes. All manual integrations receive a second level review.

Figure 20-1. Example - Demonstration of Capability Documentation

Laboratory Name:_____

Laboratory Address:_____

Method:_____ Matrix:_____

Date:_____ Analyst(s):_____

Source of Analyte(s):_____

Analytical Results

Analyte	Conc. (units)	Rep 1	Rep 2	Rep 3	Rep 4	Average % Recovery	%RSD

% RSD = Percent relative standard deviation = standard deviation divided by average % Recovery

Raw data reference: _____

Certification Statement:

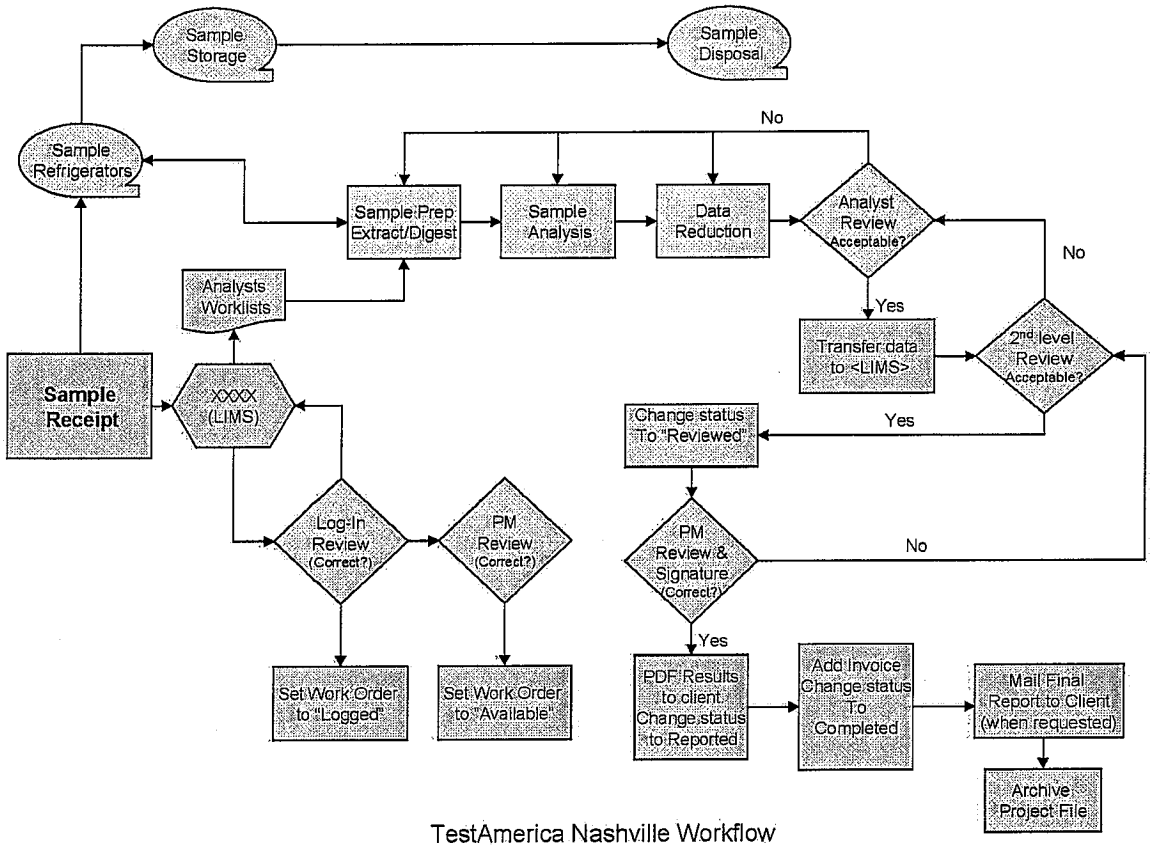
We, the undersigned, certify that:

1. The cited test method has met Demonstration of Capability requirements.
2. The test method was performed by the analyst(s) identified on this certification.
3. A copy of the test method and the laboratory-specific SOPs are available for all personnel on site.
4. The data associated with the method demonstration of capability are true, accurate, complete, and self-explanatory.
5. All raw data necessary to reconstruct and validate these analyses have been retained at the facility, and the associated information is well organized and available for review.

_____ _____
Analyst Signature Date

_____ _____
Technical Director Signature Date

Figure 20-2. **Work Flow**



TestAmerica Nashville Workflow

SECTION 21

EQUIPMENT (AND CALIBRATIONS)
(NELAC 5.5.5)

21.1 OVERVIEW

TestAmerica purchases the most technically advanced analytical instrumentation for sample analyses. Instrumentation is purchased on the basis of accuracy, dependability, efficiency and sensitivity. Each laboratory is furnished with all items of sampling, preparation, analytical testing and measurement equipment necessary to correctly perform the tests for which the laboratory has capabilities. Each piece of equipment is capable of achieving the required accuracy and complies with specifications relevant to the method being performed. Before being placed into use, the equipment (including sampling equipment) is calibrated and checked to establish that it meets its intended specification. The calibration routines for analytical instruments establish the range of quantitation. Calibration procedures are specified in laboratory SOPs. A list of laboratory equipment and instrumentation is presented in Table 21-1.

Equipment is only operated by authorized and trained personnel. Manufacturers' instructions for equipment use are readily accessible to all appropriate laboratory personnel.

21.2 PREVENTIVE MAINTENANCE

21.2.1 TestAmerica Nashville follows a well-defined maintenance program to ensure proper equipment operation and to prevent the failure of laboratory equipment or instrumentation during use. This program of preventive maintenance helps to avoid delays due to instrument failure.

21.2.2 Routine preventive maintenance procedures and frequency, such as lubrication, cleaning, and replacements, should be performed according to the procedures outlined in the manufacturer's manual. Qualified personnel must also perform maintenance when there is evidence of degradation of peak resolution, a shift in the calibration curve, loss of sensitivity, or failure to continually meet one of the quality control criteria.

21.2.2.1 Calibrations, routine maintenance, and adjustments are part of the analysts' and Department supervisors' responsibilities. However, service contracts may be in place for some instruments to cover any major repairs.

21.2.2.2 High purity gases, reagents, and spare parts are kept on hand to minimize repair time and optimize instrument performance.

21.2.3 Table 21-2 lists examples of schedules for routine maintenance. It is the responsibility of each Department supervisor to ensure that instrument maintenance logs, containing records of both preventive and corrective maintenance, are kept for all equipment in his/her department. Preventative maintenance procedures may also be outlined in analytical SOPs or instrument manuals.

21.2.4 Instrument maintenance logs are controlled and are used to document instrument problems, instrument repair and maintenance activities. Maintenance logs must be kept for all

major pieces of equipment. Instrument maintenance logs are also used to specify instrument parameters.

21.2.4.1 Documentation must include all major maintenance activities such as contracted preventive maintenance and service and in-house activities such as the replacement of electrical components, lamps, tubing, valves, columns, detectors, cleaning and adjustments.

21.2.4.2 Each entry in the instrument log includes the Analyst's initials, the date, a detailed description of the problem (or maintenance needed/scheduled), a detailed explanation of the solution or maintenance performed, and a verification that the equipment is functioning properly (state what was used to determine a return to control. e. g., CCV run on *"date"* was acceptable, or instrument recalibrated on "date" with acceptable verification, etc.).

21.2.4.3 When maintenance or repair is performed by an outside agency, service receipts detailing the service performed can be affixed into the logbooks adjacent to pages describing the maintenance performed. This stapled in page must be signed across the page entered and the logbook so that it is clear that a page is missing if only half a signature is found in the logbook.

21.2.5 In addition, the maintenance records contain:

- The identification of the instrument/equipment (instrument's Serial Number and Model Number).
- Any maintenance procedures and frequency or a reference to their location.

21.2.6 If an instrument requires repair (subjected to overloading or mishandling, gives suspect results, or otherwise has shown to be defective or outside of specified limits) it must be taken out of operation and tagged as out of service or otherwise isolated until such a time as the repairs have been made and the instrument can be demonstrated as operational by calibration and/or verification or other test to demonstrate acceptable performance. The laboratory must examine the effect of this defect on previous analyses.

21.2.7 In the event of equipment malfunction that cannot be resolved, service must be obtained from the instrument vendor manufacturer or a qualified service technician, if such a service can be tendered. If on-site service is unavailable, arrangements are made to have the instrument shipped back to the manufacturer for repair. Back up instruments, which have been approved for the analysis, perform the analysis normally carried out by the malfunctioning instrument. If the back up is not available and the analysis cannot be carried out within the needed timeframe, the samples are subcontracted.

If an instrument is sent out for service or transferred to another facility, it must be recalibrated and verified (including new initial MDL study) prior to return to lab operations.

21.3 SUPPORT EQUIPMENT

This section applies to all devices that may not be the actual test instrument, but are necessary to support laboratory operations. These include but are not limited to: balances, ovens, refrigerators, freezers, incubators, water baths, temperature measuring devices,

thermal/pressure sample preparation devices and volumetric dispensing devices if quantitative results are dependent on their accuracy, as in standard preparation and dispensing or dilution into a specified volume. A summary of support equipment requiring calibration checks can be found in Table 21-3. All raw data records associated with the support equipment are retained to document instrument performance; these include:

- instrument model number or a unique lab identification.
- identification of standards used for the calibration check.
- performance tolerances.
- results of the calibration checks, the initials of the individual making the check, and the date of the check.

21.4 INSTRUMENT CALIBRATIONS

Calibration of analytical instrumentation is essential to the production of quality data. Strict calibration procedures are followed for each method. These procedures are designed to determine and document the method detection limits, the working range of the analytical instrumentation and any fluctuations that may occur from day to day.

Sufficient raw data records are retained to allow an outside party to reconstruct all facets of the initial calibration. Records contain, but are not limited to, the following: calibration date, method, instrument, analyst(s) initials or signatures, analysis date, analytes, concentration, response, type of calibration (Avg RF, curve, or other calculations that may be used to reduce instrument responses to concentration.)

Sample results must be quantitated from the initial calibration and may not be quantitated from any continuing instrument calibration verification unless otherwise required by regulation, method or program.

If the initial calibration results are outside of the acceptance criteria, corrective action is performed and any affected samples are reanalyzed if possible. If the reanalysis is not possible, any data associated with an unacceptable initial calibration must be reported with appropriate data qualifiers (refer to Section 13).

Note: Instruments are calibrated initially and as needed after that and at least annually.

21.4.1 CALIBRATION STANDARDS

Calibration standards are prepared using the procedures indicated in the Reagents and Standards section of the determinative method SOP; however, the general procedures are described below.

21.4.1.1 For each analyte and surrogate (if applicable) of interest, prepare calibration standards at the minimum number of concentrations as stated in the analytical methods. If a reference or mandated method does not specify the number of calibration standards, the minimum number is three, not including blanks or a zero standard. All of the standard solutions are prepared using Class A volumetric glassware, calibrated pipettes, and/or microsyringes and appropriate laboratory quality solvents and stock standards.

21.4.1.2 Standards for instrument calibration are obtained from a variety of sources. All standards are traceable to national or international standards of measurement, or to national or international standard reference materials, whenever possible. Dilution standards are prepared from stock standards purchased from commercial suppliers. A standard log is maintained in LIMS by each department, containing concentration, date of receipt, date of standard preparation, any dilutions made, lot number, supplier, type of solvent and a unique code number to identify the standard.

21.4.1.3 The lowest concentration calibration standard that is analyzed during an initial calibration must be at or below the stated reporting limit for the method based on the final volume of extract (or sample).

21.4.1.4 The other concentrations define the working range of the instrument/method or correspond to the expected range of concentrations found in actual samples that are also within the working range of the instrument/method. Results of samples not bracketed by initial instrument calibration standards (within calibration range to 3 significant figures) must be reported as having less certainty, e.g., defined qualifiers or flags (additional information may be included in the case narrative). The lowest calibration standard must be at or below the reporting limit. The exception to these rules is ICP methods or other methods where the referenced method does not specify two or more standards.

21.4.1.5 Given the number of target compounds addressed by some of the organic methods, it may be necessary to prepare several sets of calibration standards, each set consisting of the appropriate number of solutions at different concentrations. The initial calibration then involves the analysis of each of these sets of the appropriate number of standards.

21.4.1.6 All initial calibrations are verified with a standard obtained from a second source and traceable to a national standard, when available (or vendor certified different lot if a second source is not available). For unique situations, such as air analysis where no other source or lot is available, a standard made by a different analyst would be considered a second source. This verification occurs immediately after the calibration curve has been analyzed, and before the analysis of any samples.

21.4.1.7 A new calibration curve must be made yearly, at minimum. All curves must document the date created.

21.4.2 CALIBRATION FOR ORGANIC METHODS (GC, HPLC, GC/MS)

21.4.2.1 Many of the organic analytical methods utilize an internal standard calibration (GCMS and some GC). Because of the complex nature of the multipeak chromatograms produced by the method, some instruments necessitate the use of external standard calibration (most GC and HPLC). Surrogate compounds are included in the calibration processes for all appropriate organic analyses. For more details on the calibration types listed below, refer to SOP No. CA-Q-S-005, Calibration Curves.

21.4.2.2 Once the operating parameters have been established according to the method, each instrument is calibrated for the appropriate method. The analyst prepares five or more standard solutions at various concentrations containing all of the analytes of interest, internal standards, and surrogates that are appropriate for the method. Note: There are a several EPA methods that have different requirements and are exceptions where a minimum of three calibration standards are prepared and analyzed.

21.4.2.3 The standard solutions are introduced into the instrument in the same manner as samples are; whether it is by direct injection, by headspace analysis, or by purge and trap. The calibration factor (CF) for methods that use external standards, and the response factor (RF) for methods that use internal standards are calculated for the five (or more) standards.

- External standard calibration involves comparison of instrument responses from the sample to the responses from the target compounds in the calibration standards. Sample peak areas (or peak heights) are compared to peak areas (or heights) of the standards. The ratio of the response to the amount of analyte in the calibration standard is defined as the Calibration factor (CF). Note: The calibration models in section 21.4.6.7 or 21.4.6.8 are used for Ion Chromatography (Average CF is not used.)

- Internal standard calibration involves the comparison of instrument responses from the target compounds in the sample to the responses of specific standards added to the sample or sample extract prior to injection. The ratio of the peak area (or height) of the target compound in the sample or sample extract to the peak area (or height) of the internal standard in the sample or sample extract is compared to a similar ratio derived for each calibration standard. The ratio is termed the response factor (RF), and may also be known as a relative response factor in other methods.

In many cases, internal standards are recommended. These recommended internal standards are often brominated, fluorinated, or stable isotopically labeled analogs of specific target compounds, or are closely related compounds whose presence in environmental samples is highly unlikely. The use of specific internal standards is available in the method SOP.

Whichever internal standards are employed, the analyst needs to demonstrate that the measurement of the internal standard is not affected by method analytes and surrogates or by matrix interferences. In general, internal standard calibration is not as useful for GC and HPLC methods with non-MS detectors because of the inability to chromatographically resolve many internal standards from the target compounds. The use of MS detectors makes internal standard calibration practical because the masses of the internal standards can be resolved from those of the target compounds even when chromatographic resolution cannot be achieved.

When preparing calibration standards for use with internal standard calibration, add the same amount of the internal standard solution to each calibration standard, such that the concentration of each internal standard is constant across all of the calibration standards, whereas the concentrations of the target analytes vary. The internal standard solution contains one or more internal standards and the concentration of the individual internal standards may differ within the spiking solution (e. g., not all internal standards need to be at the same

concentration in this solution). The mass of each internal standard added to each sample extract immediately prior to injection into the instrument or to each sample prior to purging must be the same as the mass of the internal standard in each calibration standard. Calculate the volume of the solution spiked into sample extracts such that minimal dilution of the extract occurs (e. g., 10 uL of solution added to a 1 mL final extract results in only a negligible 1% change in the final extract volume which can be ignored in the calculations).

An ideal internal standard concentration would yield a response factor of 1 for each analyte. However, this is not practical when dealing with more than a few target analytes. Therefore, as a general rule, the amount of internal standard produces an instrument response (e. g., area counts) that is no more than 100 times that produced by the lowest concentration of the least responsive target analyte associated with the internal standard. This results in a minimum response factor of approximately 0.01 for the least responsive target compound. Refer to SOP No. CA-Q-S-005, Calibration Curves, for specific calculations.

Calibration Factors and Response Factors for each analyte are calculated as follows:

$$\textbf{Calibration Factor (CF)} = \frac{A(s)}{C(s)}$$

$$\textbf{Response Factor (RF)} = \frac{A(s) \times C(is)}{A(is) \times C(s)}$$

where
A(s) = Peak area (or height) of the analyte or surrogate.
A(is) = Peak area (or height) of the internal standard.
C(s) = Concentration (or mass) of the analyte or surrogate, in ug/L.
C(is) = Concentration (or mass) of the internal standard, in ug/L.

Note: In the equation above, RF is unit-less, i. e., the units from the two area terms and the two concentration terms cancel out. Therefore, units other than ug/L may be used for the concentrations of the analyte, surrogate, and internal standard, provided that both C(s) and C(is) are expressed in the same units. The mass of the analyte and internal standard may also be used in calculating the RF value.

The CF or RF for each analyte at each concentration is tabulated to determine the graphical linearity of concentration versus response factor or calibration factor. The five (or more) CFs or RFs for each analyte in the initial calibration must have an acceptable Percent Relative Standard Deviation (% RSD) that is determined by each analytical method. If the RSD of the calibration or response factors is less than or equal to the acceptance limit stated in the published method over the calibration range, then linearity through the origin may be assumed, and the average calibration response factor may be used to determine sample concentrations. The CFs or RFs for each compound are calculated and kept in the calibration files.

The % Relative Standard Deviation is calculated as follows:

$$\%RSD = (SD / \overline{x_i}) \times 100$$

where SD = Standard Deviation of initial 5 CFs or RFs for each compound calculated as follows:

$$SD = \sqrt{\sum_{i=1}^{n} \frac{\left(x_i - \overline{x_i}\right)^2}{n-1}}$$

where

$\overline{x_i}$ = Mean (Average) of initial 5 CFs or RFs for each compound.

n = number of standards

x_i = individual CF or RF

21.4.2.4 Policies regarding the use of calibration standard results for creating the calibration curve are as follows:

- A low calibration standard may be excluded from the calibration if the signal-to-noise ratio or spectral criteria are not suitable. The reporting limit must be elevated to be the lowest calibration standard used for calibration.
- The upper calibration standard may be excluded if it saturates the detector or is obviously becoming non-linear. Any sample exceeding the upper standard used in the calibration must be diluted and re-analyzed.
- Mid-calibration standards may not be excluded unless an obvious reason is found, i. e., cracked vial, incorrectly made, etc. The failed standard may be re-run immediately (within 24 hours of the calibration run and prior to the processing of samples against the curve) and inserted into the initial calibration. If not useful, recalibration is required.

21.4.2.5 Percent RSD Corrective Action

Given the potentially large numbers of analytes that may be analyzed in some methods, it is likely that some analytes may exceed the acceptance limit for the RSD for a given calibration. In those instances, the following steps are recommended, but not required.

21.4.2.5.1 The first step is generally to check the instrument operating conditions. This option applies in those instances where a linear instrument response is expected. It may involve some trade-offs to optimize performance across all target analytes. For instance, changes to the operating conditions necessary to achieve linearity for problem compounds may cause the RSD for other compounds to increase, but as long as all analytes meet the RSD limits for linearity, the calibration is acceptable.

21.4.2.5.2 If the RSD for any analyte is greater than the applicable acceptance criteria in the applicable analytical method SOP, the analyst may wish to review the results (area counts, calibration or response factors, and RSD) for those analytes to

ensure that the problem is not associated with just one of the initial calibration standards. If the problem appears to be associated with a single standard, that one standard may be reanalyzed and the RSD recalculated. Replacing the standard may be necessary in some cases.

21.4.2.5.3 A third alternative is to narrow the calibration range by replacing one or more of the calibration standards with standards that cover a narrower range. If linearity can be achieved using a narrower calibration range, document the calibration linearity, and proceed with analyses. Changes to the upper end of the calibration range affect the need to dilute samples above the range, while changes to the lower end affect the overall sensitivity of the method. Consider the regulatory limits or action levels associated with the target analytes when adjusting the lower end of the range.

Note: When the purpose of the analysis is to demonstrate compliance with a specific regulatory limit or action level, the laboratory must ensure that the method quantitation limit is at least as low as the regulatory limit or action level.

21.4.2.6 Alternatively, the least squares regression may be used to determine linearity. A five point line must result in a correlation coefficient (r) of 0.995 or better ($r^2 \geq 0.99$) using the least squares method to be considered acceptable. In some cases it may be allowable that the curves be forced through zero (not to be confused with including the origin as an additional data point, which is not allowed). **Note:** EPA Method 8000B does not allow forcing through zero; however, the agency has revaluated this position and has since changed this stance to allow forcing through zero. In addition, from EPA Method 8000C: "However, the use of a linear regression or forcing the regression through zero may NOT be used as a rationale for reporting results below the calibration range demonstrated by the analysis of the standards." **OH VAP does not allow forcing through zero on any samples taken for the purpose of their program. South Carolina does not allow forcing through zero for the linear calibration curve.**

21.4.2.7 Instead of a linear curve model (either Average RF or least squares regression), a second order curve (quadratic) may be used (and preferred) as long as it contains at least six data points. As a rule of thumb, if there is a consistent trend in RFs (or CFs) in the calibration curve, either up or down, then quadratic curve fit may be indicated as the preferred calibration routine for that analyte. The coefficient of determination (COD or r^2) for the quadratic curve must be at least 0.99 for it to be considered acceptable. For more details on the calculations, see Calibration Curve SOP CA-Q-S-005. Some limitations on the use of quadratic curve fits:

21.4.2.7.1 Care MUST be exercised to assure that the results from this equation are real, positive, and fit the range of the initial calibration.

21.4.2.7.2 They **must not** be used to mask instrument problems that can be corrected by maintenance. (Not to be used where the analyte is normally found to be linear in a properly maintained instrument).

21.4.2.7.3 They **must not** be used to compensate for detector saturation. If it is suspected that the detector is being saturated at the high end of the curve, remove the higher concentration standards from the curve and try a 1st order fit or average RF.

Coefficient of Determination

$$r^2 = \frac{\left(\sum xy\right)^2}{\sum x^2 \sum y^2}$$

Correlation Coefficient

$$r = \frac{\left(\sum xy\right)}{\sqrt{\sum x^2 \sum y^2}}$$

where:

y = Response or Response ratio (see below)

x = Concentration

21.4.2.7.4 South Carolina does not allow the use of second order regression. Second order (quadratic) curve fits may not be used when analyzing any South Carolina samples.

21.4.3 Calibration for Inorganic Analyses

EPA Method 7000 from EPA SW-846 is a general introduction to the quality control requirements for metals analysis. For inorganic methods, quality control measures set out in the individual methods and in the *Standard Methods for the Examination of Water and Wastewater* (20th Edition) may also be included. Standard Operating Procedures for the analysis and the quality control documentation measures are kept in the departments' SOP reference binders and the Control Limits Manual.

In general, inorganic instrumentation is calibrated with external standards. Some exceptions would be Inductively Coupled Plasma (ICP), Inductively Coupled Plasma Mass Spec (ICPMS), and Ion Chromatography Mass Spec (ICMS). These analyses may use an internal standard to compensate for viscosity or other matrix effects. While the calibration procedures are much the same for inorganics as they are for organics, CF's or RF's are not used. The calibration model in 21.4.2.6 is generally used for most methods, however in some instances the model from section 21.4.2.7 may be used. A correlation coefficient (r) of 0.995 or greater must be used to accept a calibration curve generated for an inorganic procedure. Correlation coefficients are determined by hand-held scientific calculators or by computer programs and documented as part of the calibration raw data. Coefficients of calibration curves used for quantitation must be documented as part of the raw data. Curves are not allowed to be stored in calculator memories and must be written on the raw data for the purposes of data validation.

21.4.3.1 "Calibrations" for titrimetric analyses are performed by standardizing the titrants against a primary standard solution or certified solutions, as applicable. See specific methods in *Standard Methods for the Examination of Water and Wastewater* (18 and 20th Edition) for more information.

21.4.3.2 Spreadsheets that are used for general chemistry calculations must have all cells containing calculations locked to prevent accidental changes to the calculations.

21.4.3.3 Instrument technologies (e. g., ICP) with validated techniques from the instrument manufacturer or other methods using a zero point and single point calibration require the following:

21.4.3.3.1 The instrument is calibrated using a zero point and a single point calibration standard.

21.4.3.3.2 The linear range is established by analyzing a series of standards, one at the reporting limit (RL).

21.4.3.3.3 Sample results within the established linear range do not need to be qualified.

21.4.3.3.4 The zero point and single standard is run daily with each analytical batch.

21.4.3.3.5 A standard at the RL is analyzed daily with each analytical batch and must meet established acceptance criteria.

21.4.3.3.6 The linearity is verified at a frequency established by the manufacturer or method.

21.4.4 Calibration Verification

The calibration relationship established during the initial calibration must be verified at periodic intervals as specified in the laboratory method SOPs in accordance with the referenced analytical methods and NELAC (2003) standard, Section 5.5.5.10. The process of calibration verification applies to both external standard and internal standard calibration techniques, as well as to linear and non-linear calibration models.

Note: The process of calibration verification referred to is fundamentally different from the approach called "calibration" in some methods. As described in those methods, the calibration factors or response factors calculated during calibration are used to update the calibration factors or response factors used for sample quantitation. This approach, while employed in other EPA programs, amounts to a daily single-point calibration, and is not appropriate nor permitted in SW-846 chromatographic procedures for trace environmental analyses.

All target analytes and surrogates, including those reported as non-detects, must be included in periodic calibration verifications for purposes of retention time confirmation and to demonstrate that calibration verification criteria are being met.

All samples must be bracketed by periodic analyses of standards that meet the QC acceptance criteria (e. g., calibration and retention time). The frequency is found in the determinative methods or SOPs.

Note: If an internal standard calibration is being used (basically GCMS) then bracketing standards are not required, only daily verifications are needed. The results from these verification standards must meet the calibration verification criteria and the retention time criteria (if applicable).

21.4.4.1 Generally, the initial calibrations must be verified at the beginning of each 12-hour analytical shift during which samples are analyzed. (Some methods may specify more or less frequent verifications). The 12-hour analytical shift begins with the injection of the calibration verification standard (or the MS tuning standard in MS methods). The shift ends after the completion of the analysis of the last sample or standard that can be injected within 12 hours of the beginning of the shift.

21.4.4.2 A continuing instrument calibration verification (CCV) must be repeated at the beginning and at the end of each analytical batch for non-GC/MS methods. Some methods have more frequent CCV requirements see specific SOPs. Most Inorganic methods require the CCV to be analyzed after ever 10 samples.

21.4.4.3 The acceptance limits for calibration verifications can be found in each method SOP. As a rule of thumb: GCMS $\pm$ 20%, GC and HPLC $\pm$ 15%, Inorganics: $\pm$ 10 or 15%. Actual methods may have wider or tighter limits; see the method SOP for specifics.

21.4.4.4 If the response (or calculated concentration) for an analyte is within the acceptance limits of the response obtained during the initial calibration, then the initial calibration is considered still valid, and the analyst may continue to use the CF, RF or % drift values from the initial calibration to quantitate sample results.

21.4.4.5 If the response (or calculated concentration) for any analyte varies from the mean response obtained during the initial calibration by more than the acceptance criteria, then the initial calibration relationship may no longer be valid. If routine corrective action procedures fail to produce a second, consecutive (immediate) calibration verification within acceptance criteria, then either the laboratory has to demonstrate performance after corrective action with two, consecutive, successful, calibration verifications, or a new, initial, instrument calibration must be performed. However, sample data associated with an unacceptable, calibration verification may be reported as qualified data under the following special conditions:

21.4.4.5.1 When the acceptance criteria for the calibration verification are exceeded high, i. e., high bias, and there are associated samples that are non-detects, then those non-detects may be reported. Otherwise, the samples affected by the unacceptable calibration verification must be reanalyzed after a new calibration curve has been established, evaluated and accepted.

21.4.4.5.2 When the acceptance criteria for the calibration verification are exceeded low, i. e., low bias, those sample results may be reported if they exceed a maximum regulatory limit/decision level. Otherwise, the samples affected by the unacceptable verification must be reanalyzed after a new calibration curve has been established, evaluated and accepted.

21.4.4.6 Verification of Linear Calibrations

Calibration verification for linear calibrations involves the calculation of the percent drift or the percent difference of the instrument response between the initial calibration and each subsequent analysis of the verification standard. Use the equations below to calculate % Drift or % Difference, depending on the procedure specified in the method SOP. Verification

standards are evaluated based on the % Difference from the average CF or RF of the initial calibration or based on % Drift or % Recovery if a linear or quadratic curve is used.

The Percent Difference is calculated as follows:

$$\% \text{ Difference} = \frac{(\text{CF(v) or RF(v)}) - (\text{Avg. CF or RF})}{(\text{Avg. CF or RF})} \times 100$$

where:

CF(v) or RF(v) = CF or RF from verification standard

Avg. CF or RF = Average CF or RF from Initial Calibration.

The Percent Drift is calculated as follows:

$$\% \text{ Drift} = \frac{\text{Result - True Value}}{\text{True Value}} \times 100$$

The Percent Recovery is calculated as follows:

$$\% \text{ Recovery} = \frac{\text{Result}}{\text{True Value}} \times 100$$

21.4.4.7 Verification of a Non-Linear Calibration

Calibration verification of a non-linear calibration is performed using the percent drift or percent recovery calculations described in 21.4.4.6 above.

Regardless of whether a linear or non-linear calibration model is used, if initial verification criterion is not met, then no sample analyses may take place until the calibration has been verified or a new initial calibration is performed that meets the specifications listed in the method SOPs. If the calibration cannot be verified after the analysis of a single verification standard, then adjust the instrument operating conditions and/or perform instrument maintenance, and analyze another aliquot of the verification standard. If the calibration cannot be verified with the second standard, then a new initial calibration is performed.

21.5 TENTATIVELY IDENTIFIED COMPOUNDS (TICS) – GC/MS ANALYSIS

For samples containing components not associated with the calibration standards, a library search may be made for the purpose of tentative identification. The necessity to perform this type of identification is determined by the purpose of the analyses being conducted. Data system library search routines do not use normalization routines that would misrepresent the library or unknown spectra when compared to each other.

Note: If the TIC compound is not part of the client target analyte list but is calibrated by the laboratory and is both qualitatively and/or quantitatively identifiable, it must not be reported as a TIC. If the compound is reported on the same form as true TICs, it must be qualified and/or narrated that the reported compound is qualitatively and quantitatively (if verification in control) reported compared to a known standard that is in control (where applicable).

For example, the RCRA permit or waste delisting requirements may require the reporting of non-target analytes. Only after visual comparison of sample spectra with the nearest library searches may the analyst assign a tentative identification.

21.5.1 Use the following guidelines for making tentative identifications

21.5.1.1 Relative intensities of major ions in the reference spectrum (ions greater than 10% of the most abundant ion) are present in the sample spectrum.

21.5.1.2 The relative intensities of the major ions must agree within ± 20%. (Example: For an ion with an abundance of 50% in the standard spectrum, the corresponding sample ion abundance must be between 30 and 70%).

21.5.1.3 Molecular ions present in the reference spectrum should be present in the sample spectrum.

21.5.1.4 Ions present in the sample spectrum but not in the reference spectrum are reviewed for possible background contamination or presence of co-eluting compounds.

21.5.1.5 Ions present in the reference spectrum but not in the sample spectrum are reviewed for possible subtraction from the sample spectrum because of background contamination or co-eluting peaks. Data system library reduction programs can sometimes create these discrepancies.

The concentration of any non-target analytes identified in the sample (see above) are estimated. The same formulae as calibrated analytes is used with the following modifications: The areas A_s and A_{is} are from the total ion chromatograms, and the RF for the compound is assumed to be 1.

The resulting concentration is reported indicating: (1) that the value is an estimate, and (2) which internal standard was used to determine concentration. Use the nearest internal standard free of interferences.

Note: The above guidelines above are from EPA SW846 III edition, method 8260B.

For general reporting if TICs are requested, the 10, largest non-target analyte peaks whose area count exceeds 10% of the nearest internal standard are termed "Tentatively Identified Compounds" (TICs). More or fewer TICs may be identified based on client requirements.

21.5.2 **TIC Reporting Limits** (RLs)

In general RLs cannot be specified because of the unknown nature of the TIC. Any reporting limit that is reported can only be evaluated as an estimate as the quantitation is based on the assumption that the TIC responds exactly as the IS responds which is most likely not the case. In general, it is not recommended to set a Reporting limit at too low of a concentration as it gives a false impression.

TICs that meet the above identification criteria (Section 21.5.1) at 10% area of the IS: The RL would be 10% of the concentration of the internal standard used for quantitation (e. g., 2.5 ug/L

for 8260B, 4.0 ug/L for 8270C). In general, if the 10% area criterion is not met, the TIC RLs are set at a level approximately five times the level of the poorest performer in the analysis.

If a compound meets the TIC criteria, the reporting limit must reflect the ratio between the TIC and the IS or five times the level of the poorest performer whichever is lower.

21.6 GC/MS TUNING

Prior to any GCMS analytical sequence, including calibration, the instrument parameters for the tune and subsequent sample analyses within that sequence must be set.

Prior to tuning/auto-tuning the mass spec, the parameters may be adjusted within the specifications set by the manufacturer or the analytical method. These generally don't need any adjustment but it may be required based on the current instrument performance. If the tune verification does not pass it may be necessary to clean the source or perform additional maintenance. Any maintenance is documented in the maintenance log.

21.6.1 The concentration of the BFB or DFTPP must be at or below the concentrations that are referenced in the analytical methods. Part of the purpose of the tune is to demonstrate sensitivity and analyzing solutions at higher concentrations does not support this purpose. Tune failures may be due to saturation and a lower BFB/DFTPP concentration may be warranted.

21.6.2 Tune evaluations usually utilize the "Autofind" function and are set up to look at the apex ±1 scan and average the three scans. Background correction is required prior to the start of the peak but no more than 20 scans before. Background correction cannot include any part of the target peak.

21.6.3 Other Options or if Auto Tune Fails:

21.6.3.1 Sometimes the instrument does not always correctly identify the apex on some peaks when the peak is not perfectly shaped. In this case, manually identify and average the apex peak ±1 scan and background correct as in Section 21.6.2 above. This is consistent with EPA 8260 and 8270.

21.6.3.2 Or the scan across the peak at one half peak height may be averaged and background corrected. This is consistent with Standard Methods 6200, EPA 624 and EPA 625.

21.6.3.3 Adjustments such as adjustments to the repeller and ion focus lenses, adjusting the EM Voltage, etc. may be made prior to tune verification as long as all of the subsequent injections in the 12 hour tune cycle are analyzed under the same MS tune settings and it is documented in the run sequence log and/or maintenance log that an adjustment was made. Excessive adjusting (more than 2 tries) without clear documentation is not allowed. Necessary maintenance is performed and documented in instrument log.

21.6.3.4 A single scan at the Apex (only) may also be used for the evaluation of the tune. For SW 846 and EPA 600 series methods, background correction is still required.

21.6.3.5 Cleaning the source or other maintenance may be performed and then follow steps for tune evaluation above. The maintenance must be documented in the maintenance log and should be noted in the sequence log. Note: If significant maintenance was performed, see methods 8000B or 8000C then the instrument may require recalibration prior to proceeding.

21.6.4 Tune evaluation printouts must include the chromatogram and spectra as well as the tune evaluation information. In addition, the verifications must be sent directly to the printer or pdf file (no screen prints for DFTPP or BFB tunes). This ability is typically built into the instrument software.

21.6.5 Since the limits are expressed in whole percentages, the results may be rounded to whole percentage before comparing to criteria when assessing the tune verification against the tune requirements. However, the comparison to the criteria is usually done automatically by the software and if the printout says "Fail" then there would have to be documentation on the raw data and comparison to the criteria if the lab intends to still accept the tune. In most cases the analyst is better off performing an adjustment and rerunning the tune standard.

21.6.6 All MS tune settings must remain constant between running the tune check and all other samples. It is recommended that a separate tune method not be used, however a separate method may be used as long as the MS conditions between the methods are the same as the sample analysis method and tracked so any changes that are made to the analysis method are also made to the tune method.

Table 21-1. Laboratory Equipment and Instrumentation

Table 21-1: Equipment/ Instrument	Manufacturer	Model Number	Serial Number	Year Put into Service	Condition When Received
ABSORPTION UNIT	MITSUBISHI	Sigma 10	7520591	1996	USED
ACCELERATED SOLVENT EXTRACTOR	DIONEX	ASE200	02060437	2002	NEW
ACCELERATED SOLVENT EXTRACTOR	DIONEX	ASE200	02060440	2002	NEW
AUTO BOAT	MITSUBISHI	ABC	75A10673	1996	USED
AUTOSAMPLER	CENTAC TECHNOLOGIES	ASX-510	120347ASX	2005	NEW
AUTOSAMPLER	AMERICAN PRECISION	3i9A	9033	1992	[1]
AUTOSAMPLER	AMERICAN PRECISION	32E-6108AY	9205	1992	[1]
AUTOSAMPLER	DIONEX	ASI-100	4290509	2005	NEW
AUTOSAMPLER	DIONEX	ASI-100	4440407	2005	NEW
AUTOSAMPLER	EST	CENTURION	CENT123020504	[1]	[1]
AUTOSAMPLER	EST	CENTURION	CENT153112904	[1]	[1]
AUTOSAMPLER	EST	CENTURION	CENT124030104	2004	[1]
AUTOSAMPLER	EST	CENTURION	CENT126030104	[1]	[1]
AUTOSAMPLER	EST	CENTURION	CENT197042106	[1]	[1]
AUTOSAMPLER	EST	CENTURION	CENT196041706	[1]	[1]
AUTOSAMPLER	EST	CENTURION	CENT171060705	[1]	[1]
AUTOSAMPLER	EST	CENTURION	CENT172060705	[1]	[1]
AUTOSAMPLER	EST	CENTURION	CENT176102105	[1]	[1]
AUTOSAMPLER	EST	CENTURION	CENT174060705	[1]	[1]
AUTOSAMPLER	EST	CENTURION	CENT198050106	[1]	[1]
AUTOSAMPLER	EST	ENCON	215081902	[1]	[1]
AUTOSAMPLER	EST	ENCON	219082202	[1]	[1]
AUTOSAMPLER	HEWLETT PACKARD	718593B	3114A25542	[1]	[1]
AUTOSAMPLER	HEWLETT PACKARD	HP7673	3247A30576	2005	[1]
AUTOSAMPLER	HEWLETT PACKARD	HP7673	3514A42318	[1]	[1]
AUTOSAMPLER	HEWLETT PACKARD	HP7673A	2843A1152	[1]	[1]
AUTOSAMPLER	HEWLETT PACKARD	HP7673	3248A33147	[1]	[1]
AUTOSAMPLER	HEWLETT PACKARD	HP7673	2546A01716	[1]	[1]
AUTOSAMPLER	HEWLETT PACKARD	HP7673	3514A42318	2007	USED
AUTOSAMPLER	SHIMADZU	SIL-10A	60341F	1997	NEW
AUTOSAMPLER	SHIMADZU	SIL-10A	C20363506886	1999	NEW
AUTOSAMPLER	SHIMADZU	SIL-10A	C20363506585	1999	NEW
AUTOSAMPLER	SHIMADZU	AOC20i	C11143602793	[1]	[1]
AUTOSAMPLER	SHIMADZU	AOC20i	C11144208289	2004	[1]
AUTOSAMPLER	SHIMADZU	AOC20i	C11143703828	[1]	[1]
AUTOSAMPLER	SHIMADZU	AOC20i	C11143703823	[1]	[1]

Table 21-1: Equipment/ Instrument	Manufacturer	Model Number	Serial Number	Year Put into Service	Condition When Received
AUTOSAMPLER	SHIMADZU	AOC20i	C11143602454	1	1
AUTOSAMPLER	SUPERIOR ELECTRIC	MO61-LF-517	A218524	1992 OR 93	1
AUTOSAMPLER	SUPERIOR ELECTRIC	MO61-LF-517	033473653	2003	1
AUTOSAMPLER	SUPERIOR ELECTRIC	MO61-LF-517	040939389	2004	1
AUTOSAMPLER	THERMO JARRELL ASH	AS300	12677003	1994	NEW
AUTOSAMPLER	THERMO JARRELL ASH	AS300	0492	1997	NEW
AUTOSAMPLER	THERMO JARRELL ASH	AS300	67247	1998	USED
AUTOSAMPLER	OI ANALYTICAL	4552	LR92489	1998	NEW
AUTOSAMPLER	VARIAN	ARCHON	11798-895	1	1
AUTOSAMPLER	VARIAN	ARCHON	12314	1998	NEW
AUTOSAMPLER	VARIAN	ARCHON	13799	2002	NEW
AUTOSAMPLER	VARIAN	ARCHON	13795	2002	NEW
AUTOSAMPLER	VARIAN	ARCHON	13735	2002	NEW
AUTOSAMPLER	VARIAN	ARCHON	13790	2002	NEW
AUTOSAMPLER	VARIAN	ARCHON	13800	2002	NEW
AUTOSAMPLER	VARIAN	ARCHON	12201	1998	NEW
AUTOSAMPLER	VARIAN	ARCHON	12194	1998	NEW
AUTOSAMPLER	VARIAN	ARCHON	12307	1998	NEW
AUTOSAMPLER	VARIAN	ARCHON	12952	1998	NEW
AUTOSAMPLER	VARIAN	ARCHON	12761	1998	NEW
AUTOSAMPLER	VARIAN	ARCHON	12974	1999	NEW
AUTOSAMPLER	VARIAN	ARCHON	13161	1	1
AUTOSAMPLER	VARIAN	ARCHON	13812	1	1
AUTOSAMPLER	VARIAN	ARCHON	13765	1	1
AUTOSAMPLER	VARIAN	ARCHON	12945	1	1
AUTOSAMPLER	VARIAN	ARCHON	13722	1	1
AUTOSAMPLER	VARIAN	ARCHON	12920	1	1
AUTOSAMPLER	VARIAN	ARCHON	13600	1	1
AUTOSAMPLER	VARIAN	ARCHON	13641	1	1
AUTOSAMPLER	VARIAN	ARCHON	13087	1997	NEW
AUTOSAMPLER	VARIAN	ARCHON	12958	1996	USED
AUTOSAMPLER	VARIAN	ARCHON	13748	1995	NEW
AUTOSAMPLER	VARIAN	ARCHON	13291	1994	NEW
AUTOSAMPLER	VARIAN	ARCHON	13077	1	1
AUTOSAMPLER	VARIAN	ARCHON	13723	2006	USED
AUTOSAMPLER	VARIAN	ARCHON	13165	1994	NEW
AUTOSAMPLER TOWER	HEWLETT PACKARD	18596C	3530A43392	1	1
AUTOSAMPLER TOWER	HEWLETT PACKARD	18596C	3120A26333	1	1
AUTOSAMPLER TOWER	HEWLETT PACKARD	18596C	3120A27415	1	1
AUTOSAMPLER TOWER	HEWLETT PACKARD	18593A	2843A1162	1	1

Table 21-1: Equipment/ Instrument	Manufacturer	Model Number	Serial Number	Year Put into Service	Condition When Received
AUTOSAMPLER TOWER	HEWLETT PACKARD	18593B	3114A25542	1	1
AUTOSAMPLER TOWER	HEWLETT PACKARD	18593B	3447A40940	1	1
AUTOSAMPLER TOWER	AGILENT	7683	US03815233	1	1
AUTOSAMPLER TOWER	AGILENT	7683	US91807060	1	1
AUTOSAMPLER TOWER	AGILENT	7683	US94209739	1	1
AUTOSAMPLER TOWER	AGILENT	7683	US00411367	1	1
AUTOSAMPLER TOWER	AGILENT	7683	CN30729442	2010	USED
AUTOSAMPLER TOWER	AGILENT	1200	DE64775625	2010	NEW
AUTOSAMPLER TRAY	HEWLETT PACKARD	18593B	3106A21456	1	1
AUTOSAMPLER TRAY	HEWLETT PACKARD	18593B	3415A35076	1	1
AUTOSAMPLER TRAY	HEWLETT PACKARD	18593B	3339A33311	1	1
AUTOSAMPLER TRAY	HEWLETT PACKARD		3348A31236	1	1
AUTOSAMPLER TRAY	HEWLETT PACKARD		(unreadable)	1	1
AUTOSAMPLER TRAY	HEWLETT PACKARD		3131A25799	1	1
AUTOSAMPLER TRAY	HEWLETT PACKARD		3304A31194	1	1
AUTOSAMPLER TRAY	AGILENT	7683	US82601220	1	1
AUTOSAMPLER TRAY	AGILENT	7683	US81100420	1	1
AUTOSAMPLER TRAY	AGILENT	7683	US24314927	1	1
AUTOSAMPLER TRAY	AGILENT	7683	US00407128	1	1
AUTOSAMPLER TRAY	AGILENT	7683	CN91653019	2010	USED
AUTOSAMPLER TRAY	AGILENT	1200	DE64775625	2010	NEW
AUTOSAMPLER WITH HEADSPACE	MARKELOV	HS9000	HS121053007	2007	NEW
AUTOSAMPLER WITH HEADSPACE	MARKELOV	HS9000	HS120053007	2007	NEW
BALANCE	DENVER INSTR.	XL610	NO113767	1	1
BALANCE	DENVER INSTR.	XE310	NO109645	1	1
BALANCE	METTLER	AE240	N28161	1993	NEW
BALANCE	METTLER	PB602-S	1118381704	1	1

Table 21-1: Equipment/ Instrument	Manufacturer	Model Number	Serial Number	Year Put into Service	Condition When Received
BALANCE	METTLER	PB602	1117421936	1	1
BALANCE	METTLER	PB602-S	1126143319	2005	NEW
BALANCE	METTLER	PG802	1115352767	1	1
BALANCE	METTLER	PB602-S	1120430697	1	NEW
BALANCE	METTLER	AE200	F15224	1	1
BALANCE	METTLER	PM600T	H19877	1	1
BALANCE	METTLER	PB302	1116383230	1	1
BALANCE	METTLER	AE200	L94571	1	1
BALANCE	METTLER	PG203-S	119331062	1	NEW
BALANCE	METTLER	AE200	J63202	1	NEW
BALANCE	METTLER	PL601-S	1202140224	1	NEW
BALANCE	METTLER	AG204	1122173241	2003	NEW
BLOCK DIGESTOR	LACHAT	BD - 46	1800229	1994	NEW
BOD INCUBATOR	FISHER	307	WB05105311	1990	NEW
BOD INCUBATOR	FISHER	FU199ERWI	FGL52777	1989	USED
BOD INCUBATOR	KENMORE	253.65802508	BA73420257	2008	NEW
BOD INCUBATOR	EQUATHERM	HA757376	TA14SLB	1992	USED
CALORIMETER	PARR	1341	5427	1986	NEW
CENTRIFUGE	BECKMAN	ALLEGRA 6	ALS08D16	2009	NEW
CENTRIFUGE	CLAY ADAMS	DYNAC III 420104	3820031	1	NEW
CENTRIFUGE	CLAY ADAMS	DYNAC III 420104	4150009	1	NEW
CHILLER	NESLAB	CFT-33	293223	1993	NEW
COD REACTOR	HACH		901103566	1990	NEW
CONDUCTIVITY METER	WTW	330i	0148004	2002	NEW
DISCRETE ANALYZER	KONELAB	AQUA 20	E0719526	2004	NEW
DISCRETE ANALYZER	KONELAB	AQUA 20	S2419226	2009	USED
DISSOLVED OXYGEN METER	YSI	5100	00B0314	2000	NEW
DISSOLVED OXYGEN METER	YSI	5100	00D0552	2000	NEW
FLASHPOINT TESTER, AUTO	PETROLAB	PMA-4	741	1997	NEW
FLASHPOINT TESTER, AUTO	PETROLAB	PMA-4	741	1997	NEW
FLASHPOINT, OPEN CUP	FISHER	--	201N0005	2002	NEW
FLASHPOINT, PENSKY-MARTENS	FISHER	13-497-5	1787	1986	NEW
FLOW INJECTION ANALYZER	LACHAT	8000	A83000234	1995	NEW
FLOW INJECTION ANALYZER	LACHAT	FIA+	A83000-1323	1999	NEW
FLOW INJECTION ANALYZER	LACHAT	8500 FIA	050600000167	2008	USED
FLOW INJECTION ANALYZER	LACHAT	8500	090400001098	2009	NEW

Table 21-1: Equipment/ Instrument	Manufacturer	Model Number	Serial Number	Year Put into Service	Condition When Received
FLUOROMETER	SEQUOIA - TURNER	450	B002262TV	1992	NEW
FURNACE	THERMOLYN	30400	10599810039263	1984	NEW
GC w/ECD/ECD	HEWLETT PACKARD	5890	2921A23313	[1]	[1]
GC w/ECD/ECD	PERKIN ELMER	AUTOSYS	610N9122009	2000	NEW
GC w/ECD/ECD	PERKIN ELMER	AUTOSYS	610N9122008	2000	NEW
GC w/ECD/ECD	PERKIN ELMER	AUTOSYS	610N01101203	2000	USED
GC w/ECD/ECD	PERKIN ELMER	AUTOSYS XL	610N9122010	2000	NEW
GC w/ECD/ECD	SHIMADZU	17A	C11123681357	1999	NEW
GC w/ECD/ECD	HEWLETT PACKARD	6890	US00011427	2000	NEW
GC w/ECD/ECD	HEWLETT PACKARD	5890	3336A61865	2006	USED
GC w/ECD/ECD	HEWLETT PACKARD	5890	3336A51644	2007	USED
GC w/ECD/ECD	HEWLETT PACKARD	7890A	CN10734082	2009	NEW
GC w/FID	HEWLETT PACKARD	5890	2728A13149	1997	USED
GC w/FID	HEWLETT PACKARD	5890	2950A27678	[1]	[1]
GC w/FID	HEWLETT PACKARD	5890	3108A34413	1999	USED
GC w/FID	HEWLETT PACKARD	5890	3235A45233	2006	USED
GC w/FID	HEWLETT PACKARD	5890	2950A26161	2006	USED
GC w/FID	HEWLETT PACKARD	5890	3203A42028	2006	USED
GC w/FID	HEWLETT PACKARD	5890/7673	3235A45233	2006	USED
GC w/FID	HEWLETT PACKARD	5890	3336A51997	2006	USED
GC w/FID	HEWLETT PACKARD	5890	3235A46759	2009	USED
GC w/FID	HEWLETT PACKARD	6890/7683	US00029922	2010	USED
GC w/FID	PERKIN ELMER	AUTOSYS	610N1040206	1992	NEW
GC w/FID	PERKIN ELMER	CLARUS 500	650N3100901	2003	NEW
GC w/FID	PERKIN ELMER	CLARUS 500	650N4031504	2004	NEW
GC w/FID	SHIMADZU	17A	C11123580493	1998	NEW
GC w/FID	SHIMADZU	17A	C11123681362	1999	NEW
GC w/FID	SHIMADZU	17A	C11123781625	1999	NEW
GC w/FID	SHIMADZU	17A	C11123781731	1999	NEW
GC w/FID	SHIMADZU	17A	C11123781531	1999	NEW
GC w/NPD/FPD	PERKIN ELMER	CLARUS 500	650N4061602	2004	NEW
GC w/PID/FID	HEWLETT PACKARD	5890	3027A29703	2001	USED
GC w/PID/FID	HEWLETT PACKARD	5890	3126A36232	1998	USED
GC w/PID/FID	HEWLETT PACKARD	5890	2908A91269	1990	USED

Table 21-1: Equipment/ Instrument	Manufacturer	Model Number	Serial Number	Year Put into Service	Condition When Received
GC w/PID/FID	HEWLETT PACKARD	5890	3133A37656	1996	USED
GC w/PID/FID	HEWLETT PACKARD	5890	3203A42091	1996	USED
GC w/PID/FID	HEWLETT PACKARD	5890	3336A58503	1998	USED
GC w/PID/FID	HEWLETT PACKARD	5890	2938A24989	1998	USED
GC w/PID/FID	HEWLETT PACKARD	5890	3336A50264	1998	USED
GC w/PID/FID	HEWLETT PACKARD	5890	2938A25493	1998	USED
GC w/PID/FID	HEWLETT PACKARD	6890	US00041322	2002	NEW
GC w/PID/FID	HEWLETT PACKARD	6890	US00041324	2002	NEW
GC w/PID/FID	HEWLETT PACKARD	6890	US00027429	2002	NEW
GC w/PID/FID	HEWLETT PACKARD	5890	3336A60618	2003	USED
GC w/PID/FID	HEWLETT PACKARD	5890	DE00003878	2003	NEW
GC w/PID/FID	HEWLETT PACKARD	6890N	US10350005	2004	NEW
GC w/PID/FID	HEWLETT PACKARD	6890N	US10350002	2004	NEW
GC w/PID/FID	HEWLETT PACKARD	5890	2950A27370	2005	USED
GC w/PID/FID	HEWLETT PACKARD	5890	3223A43282	2005	USED
GC w/PID/FID	HEWLETT PACKARD	5890	3019A28662	2005	USED
GC w/PID/FID	HEWLETT PACKARD	5890	3203A41711	2006	USED
GC w/PID/FID	HEWLETT PACKARD	5890	3203A41407	2007	USED
GC w/PID/FID	SHIMADZU	17A	C11123580601	1998	NEW
GC w/PID/FID	SHIMADZU	17A	01123681361	1999	NEW
GC w/PID/FID	TREMETRICS	9001	100035	1996	NEW
GC w/PID/FID	TREMETRICS	9001	100036	1996	NEW
GC w/PID/FID	VARIAN	3400	4035	1999	USED
GC w/TCD	SHIMADZU	8A	C10493313148	1998	[1]
GC/MS	HEWLETT PACKARD	5890II/5972	3307A00396	1997	USED
GC/MS	HEWLETT PACKARD	5890II/5971	3234A04319	1994	NEW
GC/MS	HEWLETT PACKARD	5890II/5971	2749A00081	1997	NEW
GC/MS	HEWLETT PACKARD	5890II/5972	3329A00693	1994	NEW
GC/MS	HEWLETT PACKARD	5890II5972	3501A02393	1995	NEW
GC/MS	HEWLETT PACKARD	6890/5973	US82311321	1999	USED

Table 21-1: Equipment/ Instrument	Manufacturer	Model Number	Serial Number	Year Put into Service	Condition When Received
GC/MS	HEWLETT PACKARD	6890/5973	US80210937	1999	USED
GC/MS	HEWLETT PACKARD	6890/5973	US94222434	2000	NEW
GC/MS	HEWLETT PACKARD	6890/5973N	US01140208s	2000	NEW
GC/MS	HEWLETT PACKARD	G1530A/ G1099A	US91421544	1999	NEW
GC/MS	HEWLETT PACKARD	G1530A/ G1099A	3435A01921	1998	[1]
GC/MS	HEWLETT PACKARD	6890/5973	US81221486	2000	NEW
GC/MS	HEWLETT PACKARD	6890/5973	US91921735	2000	NEW
GC/MS	HEWLETT PACKARD	6890/5973	US94212231	2000	NEW
GC/MS	HEWLETT PACKARD	6890/5973	US94211203	2000	NEW
GC/MS	HEWLETT PACKARD	6890/5973	US94260109	2001	USED
GC/MS	HEWLETT PACKARD	6890/5973N	US03340475	2001	NEW
GC/MS	HEWLETT PACKARD	6890/5973N	US03340479	2002	NEW
GC/MS	HEWLETT PACKARD	6890/5973N	US21844021	2002	NEW
GC/MS	HEWLETT PACKARD	6890/5973N	US21844025	2002	NEW
GC/MS	HEWLETT PACKARD	5890/5972	3336A57928	2004	USED
GC/MS	HEWLETT PACKARD	5890/5972	3235A44162	2005	USED
GC/MS	HEWLETT PACKARD	5890/5972	2423A01966	2005	USED
GC/MS	HEWLETT PACKARD	5890/5973	US00027481	2005	USED
GC/MS	HEWLETT PACKARD	6890/5973	4510250045	2005	USED
GC/MS	HEWLETT PACKARD	6890/5973	US00037997	2006	USED
GC/MS	HEWLETT PACKARD	6890/5973	US00037995	2006	USED
GC/MS	HEWLETT PACKARD	6890/5973	US00027177	2006	USED
GC/MS	HEWLETT PACKARD	6890/5973	US00033436	2006	USED
GC/MS	HEWLETT PACKARD	6890/5973	US00008026	2006	USED
GC/MS	HEWLETT PACKARD	6890/5973	US00022681	2006	USED
GC/MS	HEWLETT PACKARD	6890/5973	US00022412	2008	USED
GC/MS	HEWLETT PACKARD	6890/5973	US10462174	2009	NEW

Table 21-1: Equipment/ Instrument	Manufacturer	Model Number	Serial Number	Year Put into Service	Condition When Received
GC/MS w/ENTEC CONCENTRATOR	HEWLETT PACKARD	6890/5973N	US10450528	2001	NEW
GEL PERMEATION CLEANUP	O. I. ANALYTICAL	AUTOPREP 2000	E427330254	2004	NEW
HEADSPACE ANALYZER	TEKMAR	7000	91242001	2001	USED
HOTPLATES	THERMODYNE	CIMAREC 3		1991 -	NEW
HPLC	SHIMADZU	SCL-10AVP	C21013650343	1999	NEW
HPLC	DIONEX	TCC-100MSV	1280301	2005	NEW
HPLC	DIONEX	P680A LPG-4	5550503	2005	NEW
HPLC SYSTEM (ISOCRATIC)	SHIMADZU	SCL-10AVP	C21013650380	1999	NEW
HPLC	AGILENT	1200	DE64255220 DE60557139	2010	NEW
ICP - TRACE	JARRELL - ASH	61 E	472790	1997	USED
ICP – TRACE	JARRELL - ASH	61 E	295490	1994	NEW
ICP – TRACE	THERMO SCIENTIFIC	ICAP 6000 SERIES	20084103	2009	NEW
ICP/MS	HEWLETT PACKARD	7500	JP51201460	2005	NEW
ION CHROMATO-GRAPH	METROHM	732	5142	1998	NEW
ION CHROMATO-GRAPH	METROHM	732	5122	1998	NEW
ION CHROMATO-GRAPH	DIONEX	DX600	01110237	2005	USED
KARL – FISCHER TITRATOR	BRINKMAN	684	16840013	1993	NEW
MECHANICAL SHAKER	GLAS-COL	VS50012	399665	2005	NEW
MECHANICAL SHAKER	GLAS-COL	VS50012	399872	2005	NEW
MERCURY ANALYZER	LEEMAN	Hydra AA	2007	2002	NEW
MERCURY ANALYZER	LEEMAN	HYDRA AA	6008	2008	USED
MERCURY ANALYZER	LEEMAN	HYDRA II	0002	2010	NEW
MICROWAVE	CEM	MARS XPRESS 907501	MD2979	2009	NEW
MICROWAVE	CEM	MARS XPRESS 907501	MD8469	2006	NEW
MIDI-DISTILLATION	ANDREWS GLASS		MCVA1290374	1994	NEW
MIDI-DISTILLATION	WESTCO	EASY-DIST	EA-1006	1996	NEW
OVEN	BLUE M	SW11TA1	SW3034	1989	NEW
OVEN	BLUE M	SW17TA1	SW7252	1993	NEW
OVEN	BLUE M	SWIITA-1	SW3990	1	1
OVEN	FISHER	ISOTEMP 650G	803N0038	1	1
OVEN	FISHER	ISOTEMP 516G	903N0083	1	1
OVEN	FISHER	500	51100409	1	1
OVEN	NAPCO	620	(ILLEGIBLE)	1	1
OVEN	FISHER	ISOTEMP 650G	202N0036	2002	NEW

Table 21-1: Equipment/ Instrument	Manufacturer	Model Number	Serial Number	Year Put into Service	Condition When Received
OXYGEN PROBE	ORION	97-08-00		1991	NEW
pH METER	ORION	330	005043	2000	NEW
pH/mv METER	ACCUMET	10	C9002414	1994	NEW
PH/mv METER	ACCUMET	XL15	5186494	2007	NEW
pH/mv METER	CORNING	220	12351	1991	NEW
pH/mvMETER	ACCUMET	AB15	AB92331859	2009	NEW
PURGE/TRAP	EST	ENCON	217082202 E	1997	NEW
PURGE/TRAP	EST	ENCON	126112900 E	1997	NEW
PURGE/TRAP	EST	ENCON	216081902 E	1994	NEW
PURGE/TRAP	EST	ENCON	218082202 E	1994	NEW
PURGE/TRAP	EST	ENCON	215081902E/P	1	1
PURGE/TRAP	EST	ENCON	219082202E/P	1	1
PURGE/TRAP	EST	ENCON	237082022	2006	USED
PURGE/TRAP	EST	ENCON	126112900	2006	USED
PURGE/TRAP	HEWLETT PACKARD	G1900-60500	3651A10630	1995	NEW
PURGE/TRAP	HEWLETT PACKARD	3000	3636A10579	1	1
PURGE/TRAP	O.I. ANALYTICAL	4560	K811460272	1998	NEW
PURGE/TRAP	O.I. ANALYTICAL	4560	D308371	1993	NEW
PURGE/TRAP	O.I. ANALYTICAL	4560	H421460320	1994	NEW
PURGE/TRAP	TEKMAR	3000	93251007	1993	NEW
PURGE/TRAP	TEKMAR	3000	95093006	1995	NEW
PURGE/TRAP	TEKMAR	3000	99053016	2003	1
PURGE/TRAP	TEKMAR	3000	94057008	1	1
PURGE/TRAP	TEKMAR	3000	98236009	1	1
PURGE/TRAP	TEKMAR	3000	98139001	1	1
PURGE/TRAP	TEKMAR	3000	98098001	1	1
PURGE/TRAP	TEKMAR	3000	98364008	1	1
PURGE/TRAP	TEKMAR	3000	93316011	1	1
PURGE/TRAP	TEKMAR	3000	94252002	1	1
PURGE/TRAP	TEKMAR	3000	98111004	1	1
PURGE/TRAP	TEKMAR	3000	93334005	1	1
PURGE/TRAP	TEKMAR	3000	95144001	1	1
PURGE/TRAP	TEKMAR	3000	95166007	1	1
PURGE/TRAP	TEKMAR	3000	94266005	1	1
PURGE/TRAP	TEKMAR	3000	99196020	1	1
PURGE/TRAP	TEKMAR	3000	97009013	1	1
PURGE/TRAP	TEKMAR	3000	98092002	1	1
PURGE/TRAP	TEKMAR	3000	995207010	1	1
PURGE/TRAP	TEKMAR	3000	98008002	1	1
PURGE/TRAP	TEKMAR	3000	3449A10215	1	1
PURGE/TRAP	TEKMAR	3000	95153007	1	1
PURGE/TRAP	TEKMAR	3000	98229017	1	1
PURGE/TRAP	TEKMAR	3000	94005035	1	1
PURGE/TRAP	TEKMAR	3000	94005015	1	1
PURGE/TRAP	TEKMAR	3000	96325013	1	1
PURGE/TRAP	TEKMAR	3000	93170002	1	1
PURGE/TRAP	TEKMAR	3000	99076004	1	1

Table 21-1: Equipment/ Instrument	Manufacturer	Model Number	Serial Number	Year Put into Service	Condition When Received
PURGE/TRAP	TEKMAR	LCS-3000	94067010	2002	NEW
PURGE/TRAP	TEKMAR	3100	02115005	1	1
PURGE/TRAP	TEKMAR	3100	98152010	1	1
PURGE/TRAP	TEKMAR	3100	00104011	1	1
PURGE/TRAP	TEKMAR	3100	00104012	1996	USED
PURGE/TRAP	TEKMAR	3100	99305025	1	1
PURGE/TRAP	TEKMAR	3100	US01354002	2004	1
PURGE/TRAP	TEKMAR	3100	US02249003	2004	1
PURGE/TRAP	TEKMAR	3100	US02155008	1	1
PURGE/TRAP	TEKMAR	3100	US02326003	1	1
PURGE/TRAP	TEKMAR	3100	95129003	1	1
PURGE/TRAP	TEKMAR	3100	95039008	1	1
PURGE/TRAP	TEKMAR	3100	94104002	1	1
PURGE/TRAP	TEKMAR	3100	97273002	1	1
PURGE/TRAP	TEKMAR	LSC 2000	91309006	1997	NEW
PURGE/TRAP	TEKMAR	LSC2000	177003	1997	NEW
PURGE/TRAP	TEKMAR	LSC2000	90100003	1998	NEW
PURGE/TRAP	TEKMAR	LSC2000	90115009	1998	NEW
PURGE/TRAP	TEKMAR	LSC2000	88274012	1998	NEW
PURGE/TRAP	TEKMAR	LSC-2000	8826010	1988	NEW
PURGE/TRAP	TEKMAR	LSC-2000	91092004	1991	NEW
PURGE/TRAP	TEKMAR	LSC-2000	90043025	1991	NEW
PURGE/TRAP	TEKMAR	LSC-2000	92008004	1992	NEW
RECIPROCAL WATER BATH	NEW BRUNSWICK SCIENTIFIC	R 76	400364888	2003	NEW
ROTATOR	ASSOC. DESIGN	3740-48BRE		1994	NEW
SOLID PHASE EXTRACTOR	HORIZON TECHNOLOGY	SPE-3000XL	01-1200	2004	NEW
SOLVENT CONCENTRATOR	LABCORCO	NVAP	020998064F	2002	NEW
SOLVENT CONCENTRATOR	LABCORCO	NVAP	020897646F	2002	NEW
SOLVENT CONCENTRATOR	LABCORCO	NVAP	020998063F	2002	NEW
SONICATOR	SONICS & MATERIALS	VCX600	28785F	1999	NEW
SONICATOR	SONICS & MATERIALS	VCX600	19574D	1998	NEW
SONICATOR	SONICS & MATERIALS	VCX600	18683	2001	NEW
SONICATOR	SONICS & MATERIALS	QVC502	26925	1998	NEW
SONICATOR	SONICS & MATERIALS	VC501	23840F	1997	NEW
SONICATOR	SONICS & MATERIALS	VC750	31469F	2000	NEW
SONICATOR	SONICS & MATERIALS	VC750	37121G	2003	NEW
SONICATOR	SONICS & MATERIALS	VC750	31321G	2000	NEW

Table 21-1: Equipment/ Instrument	Manufacturer	Model Number	Serial Number	Year Put into Service	Condition When Received
SOXHLET EXTRACTOR, AUTOMATED	GERHARDT	MULTISTAT SX/PC	NA	2007	NEW
SOXHLET EXTRACTOR, AUTOMATED	GERHARDT	MULTISTAT SX/PC	NA	2007	NEW
SOXHLET EXTRACTOR, AUTOMATED	GERHARDT	MULTISTAT SX/PC	NA	2007	NEW
SOXHLET EXTRACTOR, AUTOMATED	GERHARDT	MULTISTAT SX/PC	NA	2007	NEW
THERMOMETER (NIST)	ERTCO	15041D	1409	2000	NEW
THERMOMETER (NIST)	ERTCO	ASTM 62C	2918	[1]	[1]
TOC	SHIMADZU	5050	29118894	1993	NEW
TOC	SHIMADZU	TOC-VCSN	H51204635241	2009	NEW
TOC	O.I. ANALYTICAL	1030W	F923730891 AUTOSAMPLER: 612788821	2009	NEW
TOC W/SOLIDS OPTION	SHIMADZU	5050A	36001341	1999	NEW
TOX	MITSUBISHI	SIGMA 10	75R00110	1998	USED
TOX	MITSUBISHI	TOX-100	A7M41934	2009	USED
TOX	MITSUBISHI	TOX100	A7M20849	1999	NEW
TURBIDIMETER	HF	DRT – 100B	602164	2000	NEW
UV/VIS	SHIMADZU	160U	28D06563	1990	NEW
UV/VIS	SHIMADZU	1601	A10753782132	2001	NEW
UV/VIS	HACH	DR/2500	030500005514	2003	NEW
ZHE PRESSURE	MILLIPORE	T316	22361028	1993	NEW
ZHE ROTATOR (4)	MILLIPORE	Z3	455TR4042	1990	NEW

[1]Although equipment is operational and calibration maintained, this information is not available.

Table 21-2. Preventive Maintenance Procedures for Laboratory Equipment

Table 21-2: Instrument/Equipment Type	Activity	Frequency
Gas Chromatograph	Change septum	As needed – record
	Check gases	Daily
	Replace or clip column	As needed – record. Rerun calibration/RT study
	Clean detector	As needed – record
	Check autosampler seals	Daily
	Clean injectors; replace liners	As needed – record
	Clean or replace PID lamp	As needed – record
	Vendor repair	As needed – record work order
HPLC or IC	Check seals for leakage	Each use
	Replace seals/valves/lamps	As needed – record
	Replace suppressor (IC only)	As needed – record
	Replace column	As needed – record. Rerun calibration/RT study
	Clean source/analyzer	As needed – record
	Vendor repair	As needed – record work order
GC/MS	Change Merlin (SVOC only)	As needed – record
	Bake trap (VOC only)	Daily
	Clean source	As needed – record
	Change vacuum pump oil	Biannually – record
	Clean injector; replace liner (SVOC only)	Daily
	Replace column	As needed – record. Rerun calibration/RT study
	Vendor repair	As needed – record work order
ICP	Torch inspection	Each use
	Clean torch and nebulizer	As needed – record
	Inspect filters	Daily
	Change filters	As needed – record
	Inspect pump tubing	Daily
	Change pump tubing	As needed – record
	Vendor repair	As needed – record work order
	Inspect contact rings	Each use
	Clean windows	Each use
	Align lamp	Each use
	Vendor repair	As needed – record work order
Mercury Analyzer	Inspect tubes and reagents	Daily
	Vendor repair	As needed – record work order
Microwave	Check power output	Weekly
	Check cleanliness and rotation	Each use
	Vendor repair	As needed – record work order
InfraRed spectrometer	Change dessicant	As needed – record
	Vendor repair	As needed – record work order

Table 21-2: Instrument/Equipment Type	Activity	Frequency
pH Meter	Clean electrode	Each use
	Inspect electrode	Each use
	Vendor repair	As needed – record work order
TCLP Extractors And Zero Headspace Extractors	Verify rotation	Each use – record
	Check for leakage	Each use
	Vendor repair	As needed – record work order
Turbidimeter	Check lamp	Each use
	Clean sample holder	Each use
	Vendor repair	As needed – record work order
UV/VIS Spectrometer	Check paper	Daily
	Clean sample compartment	As needed
	Auto-check calibration	Daily at start-up
	Wavelength calibration	Six months – record
	Vendor repair	As needed – record work order
Total Organic Carbon Analyzer	Check gas flow	Daily
	Check fluid level (IC reservoirs)	Daily
	Replace "O" rings	As needed – record
	Check needle	Each use
	Replace scrubbers (halogen and CO_2)	Yearly – record
	Replace catalyst	As needed – record
	Vendor repair	As needed – record work order
Total Organic Halogen Analyzer	Clean inlet tube	As needed
	Clean cell	Each use
	Check electrode levels	Each use
	Vendor repair	As needed – record work order
Weighing balances	Clean pan	Each use
	Check calibration	Daily – record
	Vendor repair	As needed – record work order
Temperature devices: refrigerators, incubators, evaporators, flash point tester, COD reactor, water circulator, drying ovens	Monitor temperature	Daily or when used (refrigerators 2 times per day) – record
	Vendor repair	As needed – record work order
Ultrasonic Disruptors	Clean, Tune by depressing tuning button while adjusting % output knob until lowest reading is obtained. For dual head, disconnect one horn at a time.	Each use -- record
	Vendor repair	As needed – record work order

Table 21-2 Instrument/Equipment Type	Activity	Frequency
Discrete Analyzer	Perform "start-up"	Daily
	Wash procedure	Daily
	Run water blank	Daily
	Clear daily files	Daily
	Check water and waste container; empty if needed	Weekly
	Check cuvette bin	Weekly
	Check syringe plunger tip; replace syringe	Weekly; as needed
	Run dichromate at 480 ηm, if problems with water blanks are noted.	As needed
	Save database to CD	Monthly
	Delete messages	Monthly
	Clean/lubricate incubator rod and fetcher arm	Monthly
	Replace lamp	Every 6 months

Table 21-3. Periodic Calibration

Table 21-3: Instrument	Type of Calibration/ Number of Standards	Frequency	Acceptance Limits	Corrective Action
Analytical Balance	Accuracy determined using A2LA-accredited NIST weights. Minimum of 2 standards bracketing the weight of interest. Inspected and calibrated by A2LA accredited person semi-annually.	Calibrated semi-annually. Daily calibration verification prior to use.	± 0.2%	Clean, check level, insure lack of drafts, and that unit is warmed up, recheck. If fails, call service.
Top Loading Balance	Accuracy determined using A2LA-accredited NIST weights. Minimum of 2 standards bracketing the weight of interest. Inspected and calibrated by A2LA accredited person semi-annually.	Calibrated semi-annually. Daily calibration verification prior to use.	± 0.5%	Clean. Replace.
A2LA-accredited NIST Weights	Accuracy determined by accredited weights and measurement laboratory.	1 year	As per certificate.	Replace.
NIST-Traceable Thermo-meter	Accuracy determined by A2LA-accredited weights and measurement laboratory.	1 year	As per certificate.	Replace.
Thermo-meter	Against NIST-traceable thermometer	Yearly at appropriate temperature range for intended use	± 1.2°C	Replace
Minimum-Maximum Thermo-meters	Against NIST-traceable thermometer	Yearly	± 1.5°C	Replace

Table 21-3: Instrument	Type of Calibration/ Number of Standards	Frequency	Acceptance Limits	Corrective Action
InfraRed Temperature Guns	Against NIST-traceable thermometer	Quarterly at appropriate temperature range for intended use.	± 1.5°C	Repair/replace
Dial-type Therm-ometers	Against NIST-traceable thermometer	Quarterly at appropriate temperature range for intended use.	± 1.5°C	Replace
Digital Therm-ometers	Against NIST-traceable thermometer	Quarterly	±1.2°C	Replace
Refrigerator	Temperature checked using NIST-traceable thermometer.	Daily. If out of range, check again in two hours.	2.7 ± 1.7°C	Adjust. Repair. While waiting for repair, seal door, attach "Out of Service" sign, move items to functional unit. Notify supervisor.
Freezer	Temperature checked using NIST-traceable thermometer	Daily. If out of range, check again in two hours.	(-10)-(-20)°C	Adjust. Repair. While waiting for repair, seal door, attach "Out of Service" sign, move items to functional unit. Notify supervisor.
Oven	Temperature checked using NIST-traceable thermometer.	When in use.	104 ± 1°C (drying) 180 ± 2°C (TDS)	Adjust. Replace.
Incubator	Temperature checked using NIST-traceable thermometer.	When in use.	BOD: 20 ± 1.0°C	Adjust. Replace.
Water Bath	Temperature checked using NIST-traceable thermometer.	When in use.	± 2°C	Adjust. Replace.
Volumetric Dispensing Devices (Eppendorf ® pipette, automatic dilutor or dispensing devices)	One delivery by weight. Using DI water, dispense into tared vessel. Record weight with device ID number. Note: For dispensers not used for analytical measurements, a label is applied to the device stating that it is not calibrated.	Monthly	± 2% Calculate accuracy by dividing weight by stated volume times 100 for percent.	Adjust. Replace.

Table 21-3: Instrument	Type of Calibration/ Number of Standards	Frequency	Acceptance Limits	Corrective Action
Glass Microliter Syringes	None	Accuracy must be initially de-monstrated if syringe was not received with a certifi-cate attesting to established accuracy.	± 1%	Not applicable.
Conductivity Meter	Cell impedance calibrated with three KCl standards.	Each use.	$r \geq 0.99$	Recalibrate.
Deionized Water	Check in-line conductivity meter on system with conductivity meter in Inorganics Department.	Weekly	<10 µmhos/cm^2	Record on log. Report discrepancies to Technical Director.
Barometer	Barometric pressure checked against 17025 standards	Yearly	±5 mBars	Replace.

SECTION 22

MEASUREMENT TRACEABILITY
(NELAC 5.5.6)

22.1 OVERVIEW

Traceability of measurements must be assured using a system of documentation, calibration, and analysis of reference standards. Laboratory equipment that are peripheral to analysis and whose calibration is not necessarily documented in a test method analysis or by analysis of a reference standard are subject to ongoing certifications of accuracy. At a minimum, these must include procedures for checking specifications of ancillary equipment: balances, thermometers, temperature, Deionized (DI) and Reverse Osmosis (RO) water systems, automatic pipettes and other volumetric measuring devices. With the exception of Class A Glassware (including glass microliter syringes that have a certificate of accuracy), quarterly accuracy checks are performed for all mechanical volumetric devices. Wherever possible, subsidiary or peripheral equipment is checked against standard equipment or standards that are traceable to national or international standards. Class A Glassware should be routinely inspected for chips, acid etching or deformity. If the Class A glassware is suspect, the accuracy of the glassware must be assessed prior to use.

22.2 NIST-TRACEABLE WEIGHTS AND THERMOMETERS

Reference standards of measurement are used for calibration only and for no other purpose, unless it can be shown that their performance as reference standards would not be invalidated.

For NIST-traceable weights and thermometers, the laboratory requires that all calibrations be conducted by a calibration laboratory accredited by A2LA, NVLAP (National Voluntary Laboratory Accreditation Program), APLAC (Asia-Pacific Laboratory Accreditation Cooperation), or EA (European Cooperation for Accreditation). A certificate and scope of accreditation is kept on file at the laboratory. Refer to Section 21 for calibration of weights and thermometers.

Calibration laboratory's policy for achieving measurement traceability is defined and includes the subsequent elements of uncertainty.

The uncertainty calculations of the calibration laboratory are supported by uncertainty budgets and are represented by expanded uncertainties typically using a coverage factor of k=2 to approximate the 95% confidence level. This explanation accompanies the measurement result and the associated uncertainty.

The tolerance uncertainty ratio (TUR) is calculated using the expanded uncertainty of the measurement, not the collective uncertainty of the measurement standards. A statement to this effect accompanies the TUR along with the coverage factor and confidence level.

The calibration report or certificate submitted to TestAmerica Nashville contains, in a well designed format, a traceability statement, the conditions under which the calibrations were made in the context of any potential influence, a compliance statement with an identified metrological specification and the pertinent clauses, a clearly identified record of the quantities and functional test results before and after re-calibration, and no recommendation on the calibration interval. Opinions and interpretations of results are presented along with the basis

upon which they were made and identified as such. The report may be submitted by facsimile or other electronic means as long as the requirements of the International Standard are achieved. If significant amendments are made to a calibration certificate, a supplemental certificate for the serial-number-specified piece of equipment is so identified. When a new certificate is offered, it uniquely identifies and references the one it replaces. All calibration reports are filed in the QA Office.

The calibration laboratory supports in-house calibration systems: documented procedures for in-house calibrations, evidence by a report, certificate, or sticker, for an appropriate amount of time; training records of calibration personnel; certificates from accreditation services demonstrating traceability to national or international standards of measurement; procedures for evaluating measurement uncertainty; timely and documented recalibration of reference standards. When subcontracting to a calibration laboratory, TestAmerica Nashville does not use a firm who subcontracts the work.

22.3 REFERENCE STANDARDS / MATERIALS

Reference standards/materials, where commercially available, are traceable to certified reference materials. Commercially prepared standard materials are purchased from vendors accredited by A2LA, NVLAP, NIST with an accompanying Certificate of Analysis that documents the standard purity. If a standard cannot be purchased from a vendor that supplies a Certificate of Analysis, the purity of the standard is documented by analysis. The receipt of all reference standards must be documented. Reference standards are labeled with a description of the standard, a unique Standard Identification Number and expiration date. All documentation received with the reference standard is retained as a QC record and references the Standard Identification Number.

All reference, primary and working standards/materials, whether commercially purchased or laboratory prepared, must be checked regularly to ensure that the variability of the standard or material from the 'true' value does not exceed method requirements. The accuracy of calibration standards is checked by comparison with a standard from a second source. In cases where a second standard manufacturer is not available, a vendor certified different lot is acceptable for use as a second source. For unique situations, such as air analysis where no other source or lot is available, a standard made by a different analyst would be considered a second source. The appropriate Quality Control (QC) criteria for specific standards are defined in laboratory SOPs. In most cases, the analysis of an Initial Calibration Verification (ICV) or LCS (where there is no sample preparation) is used as the second source confirmation. These checks are generally performed as an integral part of the analysis method (e.g. calibration checks, laboratory control samples).

All standards and materials must be stored and handled according to method or manufacturer's requirements in order to prevent contamination or deterioration. Refer to Table 9-1 in Section 9 for general storage requirements, and to method SOPs "Standards and Reagents" section and SOP Standards Control / NV08-214 for additional storage information. For safety requirements, please refer to method SOPs and the laboratory Environmental Health and Safety Manual.

22.4 DOCUMENTATION AND LABELING OF STANDARDS, REAGENTS, AND REFERENCE MATERIALS

Reagents must be at a minimum the purity required in the test method. The date of reagent receipt and the expiration date are documented. The lots for most of the common solvents and acids are tested for acceptability prior to company wide purchase. Refer to SOP No. CA-Q-S-001, Solvent and Acid Lot Testing and Approval.

All manufacturer or vendor supplied Certificate of Analysis or Purity must be retained, stored appropriately, and readily available for use and inspection. These records are maintained in three-ring binders in the analytical departments of the laboratory. Records must be kept of the date of receipt and date of expiration of standards, reagents and reference materials. In addition, records of preparation of laboratory standards, reagents, and reference materials must be retained, stored appropriately, and be readily available for use and inspection. For detailed information on documentation and labeling, please refer to method specific SOPs and SOP Standard Control / NV08-214.

Commercial materials purchased for preparation of calibration solutions, spike solutions, etc.., are usually accompanied with an assay certificate or the purity is noted on the label. If the assay purity is 96% or better, the weight provided by the vendor may be used without correction. If the assay purity is less than 96% a correction must be made to concentrations applied to solutions prepared from the stock commercial material.

22.4.1 All standards, reagents, and reference materials must be labeled in an unambiguous manner. Standards are logged into the laboratory's LIMS system and are assigned a unique identification number. The following information is typically recorded in the electronic database within the LIMS.

- Standard ID
- Description of Standard
- Department
- Preparer's name
- Final volume and number of vials prepared
- Solvent type and lot number
- Preparation Date
- Expiration Date
- Standard source type (stock or daughter)
- Standard type (spike, surrogate, other)
- Parent standard ID (if applicable)
- Parent Standard Analyte Concentration (if applicable)
- Parent Standard Amount used (if applicable)
- Component Analytes
- Final concentration of each analyte
- Comment box (text field)

Records are maintained electronically for standard and reference material preparation. These records show the traceability to purchased stocks or neat compounds. These records also include method of preparation, date of preparation, expiration date, and preparer's name or initials. Preparation procedures are provided in the Method SOPs.

22.4.2 All standards, reagents, and reference materials must be clearly labeled with a minimum of the following information:

- Expiration Date (include prep date for reagents)
- Standard ID (from LIMS)
- Description of standard (if different from manufacturer's label or if standard was prepared in the laboratory)
- Special Health/Safety warnings if applicable. See EH&S manual for further information.

22.4.3 In addition, the following information may be helpful:

- Date of receipt for commercially purchased items or date of preparation for laboratory prepared items
- Date opened (for multi-use containers, if applicable)
- Concentration (if applicable)
- Initials of analyst preparing standard or opening container

All containers of prepared reagents must include a preparation date, expiration date and an ID number to trace back to preparation.

Procedures for preparation of reagents can be found in the Method SOPs.

To maintain traceability, standard ID numbers must be noted on all associated logbooks, worksheets and raw data.

All reagents and standards must be stored in accordance to the following priority: 1) with the manufacturer's recommendations; 2) with requirements in the specific analytical methods as outlined in lab Method SOPs; and 3) according to SOP Standard Control / SA08-214 and Table 9-2.

SECTION 23

SAMPLING
(NELAC 5.5.7)

23.1 OVERVIEW

TestAmerica Nashville does not provide sampling services. The laboratory's responsibility in the sample collection process lies in supplying the sampler with the necessary coolers, reagent water, sample containers, preservatives, sample labels, custody seals, COC forms, ice, and packing materials required to properly preserve, pack, and ship samples to the laboratory

23.2 SAMPLING CONTAINERS

The laboratory offers clean sampling containers for use by clients. These containers are obtained from reputable container manufacturers and meet EPA specifications as required. Any certificates of cleanliness that are provided by the supplier are maintained at the laboratory.

23.2.1 Preservatives

Upon request, preservatives are provided to the client in pre-cleaned sampling containers. In some cases containers may be purchased pre-preserved from the container supplier. Whether prepared by the laboratory or bought pre-preserved, the grades of the preservatives are at a minimum:

- Hydrochloric Acid – Reagent ACS (Certified VOA Free) or equivalent
- Methanol – Purge and Trap grade
- Nitric Acid – Instra-Analyzed or equivalent
- Sodium Bisulfate – ACS Grade or equivalent
- Sodium Hydroxide – Instra-Analyzed or equivalent
- Sulfuric Acid – Instra-Analyzed or equivalent
- Sodium Thiosulfate – ACS Grade or equivalent

23.2.2 Preparing Container Orders

When new containers arrive at the laboratory, the date of receipt is recorded on the packing list received with them for retained documentation. Periodically, containers are evaluated for cleanliness based upon their intended parameter sample analysis. Upon request, the containers are then sent to clients for use in collecting samples. The shipping date, type and number of containers are maintained on file by the lab. Shipping personnel insure that container stock is rotated so that "first in" is "first out." When a client requests containers, a client services representative creates a container request in LIMS; it is then stored permanently in LIMS with a unique container order number. Copies of the container request are printed for the shipping department. One copy goes to the client with the containers; one copy is filed in the shipping department.

The laboratory also provides EnCore, TerraCore or other soil sampling devices when requested.

If containers are provided directly to the client from the manufacturer or from other sources, the laboratory will not be responsible for any of the above records.

23.3 FIELD QUALITY CONTROL (QC)

Common field quality control samples are defined in the following paragraphs. The frequency of field quality control samples should be specified in the site specific Quality Assurance Project Plan (QAPP) or by the client. TestAmerica provides trip blanks for VOC analysis with the sample containers for all volatile organic analyses. Blanks generated in the field are analyzed along with the field samples.

23.3.1 Equipment Blank / Rinsate Blank - The equipment blank, sometimes referred to as a rinsate blank, is a sample of the water used to decontaminate sampling equipment. The source water must be as free of target analytes as possible. An aliquot of this water is poured over or through the sample collection device after decontamination, collected in a sample container, preserved with appropriate reagents, and returned to the laboratory. This serves as a check on sampling device cleanliness, and are also affected by the site and sample handling conditions evaluated by the other types of blanks. The sampling time for the equipment blank begins when the equipment is rinsed and the water is collected.

23.3.2 Field Blank - The field blank is water that is as free of target analytes as possible and from the same source as the equipment blank. The water is poured into a sampling container at the sampling site, preserved with the appropriate reagents, and returned to the laboratory. This serves as a check on reagent and environmental contamination. The sampling time for the field blank is when the blank is prepared in the field.

23.3.3 Trip Blank - The trip blank pertains to volatile analysis only. This serves as a check on sample contamination originating from sample transport, sample container contamination, shipping and storage, or from certain site conditions. Trip blanks are often referred to as travel blanks. They are prepared using pre-cleaned sample containers. They are filled with organic-free water (the source of the organic free water is the same source of water used to prepare volatile standards, method blanks, LCS and sample dilutions), sealed and taken into the field with the empty containers which are to be used for sampling. Alternatively, they may be purchased as certified organic-free for targets analyzed. The recommended frequency is one trip blank per cooler (in duplicate or triplicate), per volatiles method. Unless otherwise specified, the sampling time for the trip blank is the time of receipt at the laboratory (When the "Trip" ends).

23.3.4 Field Duplicates - Field duplicates are replicate samples collected from the same sampling point or location during a field collection event. This control sample is used to demonstrate the ability of both the sampling and analytical process to generate data of acceptable precision.

23.4 DEFINITION OF HOLDING TIME

The date and time of sampling documented on the chain-of-custody (COC) form establishes the day and time zero. As a general rule, when the maximum allowable holding time is expressed in "days" (e. g., 14 days, 28 days), the holding time is based on calendar day measured. Holding

times expressed in "hours" (e. g., 6 hours, 24 hours, etc.) are measured from date and time zero. The first day of holding time ends twenty-four hours after sampling. Holding times for analysis include any necessary reanalysis. However, there are some programs that determine all holding times based on the date and specific time of sampling or to the hour, versus "days" approach.

23.4.1 Semi-Volatile - Holding times for sample preparation for semi-volatile organics are measured from the sampling date (and time where applicable) until the day (and time where applicable) solvent contacts the sample. If a sample is to be extracted on the day of expiration, the actual time of extraction must be recorded on the sample preparation worksheet. Holding times for analysis are measured from the date (and time where applicable) of initiation of extraction to the time of injection into the gas chromatograph.

23.4.2 Volatiles - Holding times for volatile organics are measured from the date (and time where applicable) of sampling to the date and time of injection into the gas chromatograph. The time of initiation of purging is considered the injection time, but data systems record the start of the chromatographic run rather than the start of purging. Hence, if a sample is so near expiration that the start-of-purging time rather than the chromatographic run time is needed to document the integrity of the sample; the analyst must observe and record the start-of-purging time in the instrument log. Extractions, e. g., for high level soils, must be completed in time to allow for analysis to be initiated within the maximum allowable holding time.

23.4.3 Inorganics - For inorganic and metals analysis, the preparation/digestion/distillation must be started within the maximum holding time as measured from the sampling date (and time where applicable).

23.5 SAMPLING CONTAINERS, PRESERVATION REQUIREMENTS, HOLDING TIMES

The preservation and holding time criteria specified in the following tables are derived from the source documents for the methods. If method required holding times or preservation requirements are not met, the reports must be qualified using a flag, footnote or case narrative. "Analyze immediately" is an EPA designation reserved for tests which, for compliance monitoring projects, are performed by field instrumentation or a laboratory "generally within 15 minutes" of sampling (Federal Register, Vol. 48, No. 209, p. 11). TestAmerica qualifies data for these parameters if analysis cannot be performed within 15 minutes of sampling. "As Soon As Possible" or "ASAP" is an EPA designation for tests for which rapid analysis is advised, but for which neither EPA nor the laboratory have a basis for a holding time.

23.6 SAMPLE ALIQUOTS / SUBSAMPLING

Taking a representative sub-sample from a container is necessary to ensure that the analytical results are representative of the sample collected in the field. The size of the sample container, the quantity of sample fitted within the container, and the homogeneity of the sample need consideration when sub-sampling for sample preparation. It is the laboratory's responsibility to take a representative subsample or aliquot of the sample provided for analysis.

Each sample is handled by analysts as if it is potentially dangerous. At a minimum, safety glasses, gloves, and lab coats must be worn when preparing aliquots for analysis.

Refer to SOP Sample Subsampling, Homogenization and Compositing / NV-08-229 for specific details on taking sample aliquots and subsampling.

SECTION 24

HANDLING OF SAMPLES
(NELAC 5.5.8)

Sample management procedures at TestAmerica Nashville ensure that sample integrity and custody are maintained and documented from sampling/receipt through disposal.

24.1 CHAIN OF CUSTODY (COC)

The COC form is the written documented history of any sample and is initiated when bottles are sent to the field or at the time of sampling. This form is completed by the sampling personnel and accompanies the samples to the laboratory where it is received and stored under the laboratory's custody. The purpose of the COC form is to provide a legal written record of the handling of samples from the time of collection until they are received at the laboratory. It also serves as the primary written request for analyses from the client to the laboratory. The COC form acts as a purchase order for analytical services when no other contractual agreement is in effect. An example of a COC form may be found in Figure 24-1.

24.1.1 Field Documentation

The information the sampler needs to provide at the time of sampling on the container label is:

- Sample identification
- Date and time
- Preservative

During the sampling process, the COC form is completed and must be legible (see Figure 24-1). This form includes information such as:

- Client name, address, phone number
- Project name and/or number
- The sample identification
- Date, time and location of sampling
- Sample collectors name
- The matrix description
- The container description
- The total number of each type of container
- Preservatives used
- Analysis requested
- Requested turnaround time (TAT)
- Any special instructions
- Purchase Order number or billing information (e. g., quote number) if available
- The date and time that each person relinquished or received the sample(s), including their signed name.

The samples are stored in a cooler with ice, as applicable, and remain solely in the possession of the client's field technician until the samples are delivered to the laboratory. The sample collector must assure that each container is in his/her physical possession or in his/her view at all times, or stored in such a place and manner to preclude tampering. The field technician relinquishes the samples in writing on the COC form to the sample control personnel at the laboratory or to a TestAmerica courier. Samples are only considered to be received by lab when personnel at the laboratory have physical contact with the samples.

Note: Independent couriers are not required to sign the COC form. The COC is usually kept in the sealed sample cooler. The receipt from the courier is stored in Sample Control by date; it lists all receipts each date.

24.1.2 Legal / Evidentiary Chain-of-Custody

If samples are identified for legal/evidentiary purposes on the COC, Sample Control completes the custody seal (Figure 24-2), retains the shipping record with the COC, and initiates an internal COC (Figure 24-3) for laboratory use by analysts and a sample disposal record (Figure 24-4).

24.2 SAMPLE RECEIPT

Samples are received at the laboratory by designated sample receiving personnel, and a unique laboratory project identification number is assigned. Each sample container is assigned a unique sample identification number that is cross-referenced to the client identification number such that traceability of test samples is unambiguous and documented. Each sample container is affixed with a durable sample identification label. Sample acceptance, receipt, tracking and storage procedures are summarized in the following sections and in full detail in SOP Sample Control / NV02-01.

24.2.1 Laboratory Receipt

When samples arrive at the laboratory, sample receiving personnel inspect the coolers and samples. The integrity of each sample must be determined by comparing sample labels or tags with the COC and by visual checks of the container for possible damage. Any non-conformance, irregularity, or compromised sample receipt must be documented on a Cooler Receipt Form, Figure 24-5.

24.2.1.1 Inspection of samples include a check for:

- Complete documentation to include sample identification, location, date and time of collection, collector's name, preservation type, sample type and any additional comments concerning the samples.
- Complete sample labels to include unique identification in indelible ink.
- Use of appropriate sample containers (see Section 23)
- Adherence to holding times as specified in the test method and/or summarized in Section 23.
- Adequate sample volume for required analyses (see Section 23).

- Damage or signs of contamination to sample container. Volatile vials are also inspected for headspace

24.2.1.2 Check and record the temperature of the samples that require thermal preservation.

- Samples are deemed acceptable if arrival temperature is just above freezing and less than or equal to 6.0°C. Samples that are hand-delivered immediately after collection may not be at the required temperatures; however, if there is evidence that the chilling process has begun, such as the arrival on ice, the samples are considered acceptable. This must be documented on the COC.

- If the samples were shipped in ice and solid ice is still present and in direct contact with samples, report the samples as "received on ice." Direct contact means samples must be surrounded by ice cubes or crushed ice. Ice present in a plastic bottle or other container does not constitute direct contact. Samples shipped with only "blue ice" may not be reported as "received on ice."

24.2.1.3 Verify sample preservation as specified in the test method. Check for correct pH as specified in the test method. The results are documented on the Cooler Receipt Form and in LIMS. In the case of volatiles, it is recorded after analysis on the run log and benchsheet. Chlorine is checked on samples requiring extractable organics, BOD, TOX, cyanide, fluoride, ammonia, TKN, CBOD and Nitrate; presence or absence is recorded.

24.2.1.4 After inspecting the samples, the sample control receiving personnel sign and date the COC form, make any necessary notes of the samples' conditions on the Cooler Receipt Form and store them in appropriate refrigerators or storage locations.

24.2.1.5 If samples are received without a COC, TestAmerica provides a generic COC form to be completed by the client when the samples are brought to the laboratory. The client is always provided with a copy of the completed COC form for their records.

24.2.1.6 If analyses with short holding times are requested, the dates and times are inspected to ensure that holding times have not already expired.

24.2.1.7 Samples received after normal working hours are left in their coolers and placed in the walk-in cooler. The person receiving the samples must record the date and time received, the presence or absence of ice and custody seals, the temperature of samples, presence and type of packing material, and initials.

24.2.1.8 Any deviations from the checks described in Section 24.2.1 that question the suitability of the sample for analysis, or incomplete documentation as to the tests required are documented using the PIPE non-conformance database (see Section 13) and are resolved by consultation with the client. If the sample acceptance criteria (Section 24.3) are not met, the laboratory must either:

- Retain all correspondence and/or records of communications with the client regarding the disposition of rejected samples, or

- Fully document any decision to proceed with sample analysis that does not meet sample acceptance criteria.

Note: North Carolina requires that they be notified when samples are processed that do not meet sample acceptance criteria.

24.2.1.9 The COC, shipping documents, documentation of any non-conformance, irregularity, or compromised sample receipt, record of client contact, and resulting instructions become part of the project record.

24.2.2 Sample Control

All samples that are received by the laboratory are logged into the LIMS to allow the laboratory to track and evaluate sample progress. Each group of samples that are logged in together (typically one project from a given client/sampling event) is assigned a unique job number. Within each job, each sampling point (or sample) receives a unique number. Sample numbers are generated sequentially over time, and are not re-assigned. A sample may be composed of more than one bottle since different preservatives may be required to perform all analyses requested. Even if multiple containers are received for a single sample, each container is uniquely identified with an alphabetic letter added to the sample number. The LIMS generates sample labels that are attached to each bottle for a given sample.

Each job/set of samples is logged into LIMS with a minimum of the following information:

- Client Name, Project Name, Address, Phone, Fax, Report to information, invoice to information (most of this information is "default information" that is stored in the LIMS).
- Date and time sampled;
- Date and time received;
- Job and/or project description, sample description;
- State or country of origin of sample. (Also, see Appendix 13, A13.7.1, for AIHA guidelines.)
- Sample matrix, special sample remarks;
- Reporting requirements (i.e., QC level, report format, invoicing format);
- Turn-around-time requirements;
- Parameters (methods and reporting limits or MDLs are default information for a given parameter)

Complete details are described in SOP Sample Control / NV02-01.

24.3 SAMPLE ACCEPTANCE POLICY

The laboratory has a written sample acceptance policy that clearly outlines the circumstances under which samples are accepted or rejected. These include:

- a COC filled out completely;
- samples must be properly labeled;
- proper sample containers with adequate volume for the analysis and necessary QC;

- samples must be preserved according to the requirements of the requested analytical method;
- sample holding times must be adhered to;
- all samples submitted for water/solid Volatile Organic analyses must have a Trip Blank submitted at the same time;
- sample container must be received in good condition;
- the project manager is notified if any sample fails to meet the criteria listed in the sample acceptance policy.

Data from samples which do not meet these criteria are flagged and the nature of the variation from policy is defined. A copy of the sample acceptance policy is provided with all laboratory-supplied container shipments.

24.4 SAMPLE STORAGE

In order to avoid deterioration, contamination or damage to a sample during storage and handling, from the time of receipt until all analyses are complete, samples are stored in refrigerators or freezers, as required. In addition, samples to be analyzed for volatile organic parameters are stored in separate refrigerators designated for volatile organic parameters only. To ensure the integrity of the samples during storage, refrigerator blanks are maintained in the volatile sample refrigerators and analyzed, at minimum, every two weeks. Samples are not stored in refrigeration units containing standards or reagents. Samples are also stored away from food and other potentially contaminating sources. Samples must not be stored in the refrigerator compartment of a unit that has standards stored in the freezer compartment.

After all analyses are complete, samples are placed into sample storage for at least 60 days. This area is at room temperature. At the end of approximately 60 days, they are disposed of in accordance with SOP Sample Disposal / NV10-83.

Access to the laboratory is controlled such that sample storage need not be locked at all times unless a project specifically demands it. Samples are accessible to laboratory personnel only. Visitors to the laboratory are prohibited from entering the refrigerator and laboratory areas unless accompanied by an employee of TestAmerica.

24.5 HAZARDOUS SAMPLES AND FOREIGN SOILS

To minimize exposure to personnel and to avoid potential accidents, hazardous and foreign soil samples are stored in designated, isolated areas. Any sample that is known to be hazardous at the time of receipt or after completion of analysis is placed in one of two refrigerators designated for highly contaminated samples. Foreign and U. S. soils, requiring separate storage (as specified in SOP USDA Disposal / NV10-162) due to potential contamination with foreign organisms, are tagged with an orange sticker, and placed in specified locations. Foreign soils are heat-treated prior to disposal.

24.6 SAMPLE SHIPPING

In the event that the laboratory needs to ship samples, the samples are placed in a cooler with enough ice to ensure the samples remain just above freezing and at or below 6.0°C during

transit. The samples are carefully surrounded by packing material to avoid breakage (yet maintain appropriate temperature). For sample shipments which include water/solid volatile organic analyses, a trip blank is enclosed when required by method specifications or state or regulatory programs. The chain-of-custody form is signed by the laboratory and attached to the shipping paperwork. Samples are generally shipped overnight express or hand-delivered by a TestAmerica courier to maintain sample integrity. All personnel involved with shipping and receiving samples must be trained to maintain the proper chain-of-custody documentation and to keep the samples intact and on ice. The Environmental, Health and Safety Manual contains additional shipping requirements.

24.7 SAMPLE DISPOSAL

Samples are retained for a minimum of 30 days after the project report is sent; however, provisions may be made for earlier disposal of samples once the holding time is exceeded. Some samples are required to be held for longer periods based on regulatory or client requirements (e. g., 60 days after project report is sent). The laboratory must follow the longer sample retention requirements where required by regulation or client agreement.

Several possibilities for sample disposal exist: the sample may be consumed completely during analysis, the sample may be returned to the customer or location of sampling for disposal, or the sample may be disposed of in accordance with the laboratory's waste disposal procedures (SOP Waste Disposal / NV10-83). All procedures in the laboratory Environmental, Health and Safety Manual are followed during disposal. Samples are normally maintained in the laboratory no longer than two months from receipt unless otherwise requested.

If a sample is part of a known litigation, the affected legal authority, sample data user, and/or submitter of the sample must participate in the decision about the sample's disposal. All documentation and correspondence concerning the disposal decision process must be kept on file. Pertinent information includes the date of disposal, nature of disposal (such as sample depletion, hazardous waste facility disposal, return to client), names of individuals who conducted the arrangements and physically completed the task. The laboratory removes or defaces sample labels prior to disposal, unless this is accomplished through the disposal method (e.g., samples are incinerated). A Waste Disposal Record (Figure 24-4) must be completed.

Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes must be stored, managed, and disposed of in accordance with all federal and state laws and regulations. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to this document and SOP Waste Disposal / NV10-83.

Figure 24-1. **Example: Chain of Custody (COC)**

Nashville Division
2960 Foster Creighton
Nashville, TN 37204

Phone: 615-726-0177
Toll Free: 800-765-0980
Fax: 615-726-3404

To assist us in using the proper analytical methods, is this work being conducted for regulatory purposes?

Compliance Monitoring? Yes____ No____

Enforcement Action? Yes____ No____

Client Name/Account #: _____

Address: _____

City/State/Zip: _____

Project Manager: _____

Telephone Number: _____ Fax No.: _____

Sampler Name: (Print) _____

Sampler Signature: _____

Site State: _____

PO#: _____

TA Quote #: _____

Project ID: _____

Project #: _____

							Preservative								Matrix						Analyze For:														RUSH TAT (Pre-Schedule)	Standard TAT	Fax Results	Send QC with report
Sample ID / Description	Date Sampled	Time Sampled	No. of Containers Shipped	Grab	Composite	Field Filtered	Ice	HNO_3 (Red Label)	HCl (Blue Label)	NaOH (Orange Label)	H_2SO_4 Plastic (Yellow Label)	H_2SO_4 Glass (Yellow Label)	None (Black Label)	Other (Specify)	Groundwater	Wastewater	Drinking Water	Sludge	Soil	Other (specify)																				

Special Instructions:

Method of Shipment: FEDEX

Laboratory Comments:
Temperature Upon Receipt:
VOCs Free of Headspace? Y N

Relinquished by:	Date	Time	Received by:	Date	Time
Relinquished by:	Date	Time	Received by TestAmerica:	Date	Time

Figure 24-2. **Example: Custody Seal**

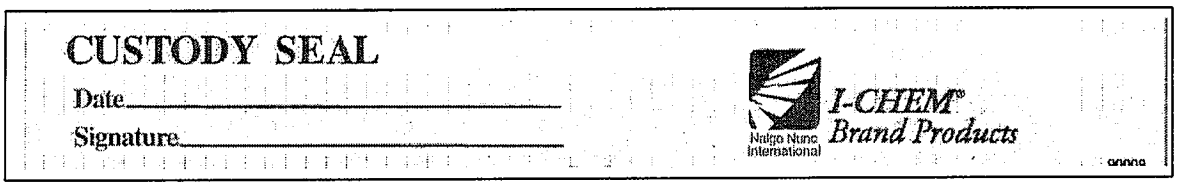


Figure 24-3. **Example: Internal Chain of Custody (COC)**

VOLATILES

Client ID	Lab Number
Influent	03-A114050
Midfluent	03-A114051
MID-BAC 1	03-A114052
MID-GAC 2	03-A114053
MID-GAC3	03-A114054
Effluent	03-A114055

Relinquished by	Received by	Reason	Date	Time

Figure 24-4. **Example: Sample Disposal Record**

Sample #	Shelf	Bottle	Day In	Day Out	Name	Disposed

Figure 24-5. **Cooler Receipt Form**

Cooler Received/Opened On_____

1. Tracking #_____(last 4 digits, FedEx)

Courier: _____ IR Gun ID_____

2. Temperature of rep. sample or temp blank when opened:_____*Degrees Celsius*

3. If Item #2 temperature is 0°C or less, was the representative sample or temp blank frozen? YES NO...NA

4. Were custody seals on outside of cooler? YES...NO...NA

If yes, how many and where:_____

5. Were the seals intact, signed, and dated correctly? YES...NO...NA

6. Were custody papers inside cooler? YES...NO...NA

I certify that I opened the cooler and answered questions 1-6 (intial) _____

7. Were custody seals on containers: YES NO and Intact YES...NO...NA

Were these signed and dated correctly? YES...NO...NA

8. Packing mat'l used? Bubblewrap Plastic bag Peanuts Vermiculite Foam Insert Paper Other None

9. Cooling process: Ice Ice-pack Ice (direct contact) Dry ice Other None

10. Did all containers arrive in good condition (unbroken)? YES...NO...NA

11. Were all container labels complete (#, date, signed, pres., etc)? YES...NO...NA

12. Did all container labels and tags agree with custody papers? YES...NO...NA

13a. Were VOA vials received? YES...NO...NA

b. Was there any observable headspace present in any VOA vial? YES...NO...NA

14. Was there a Trip Blank in this cooler? YES...NO...NA If multiple coolers, sequence #_____

I certify that I unloaded the cooler and answered questions 7-14 (intial) _____

15a. On pres'd bottles, did pH test strips suggest preservation reached the correct pH level? YES..NO..NA

b. Did the bottle labels indicate that the correct preservatives were used YES...NO...NA

If preservation in-house was needed, record standard ID of preservative used here_____

16. Was residual chlorine present? YES...NO...NA

I certify that I checked for chlorine and pH as per SOP and answered questions 15-16 (intial) _____

17. Were custody papers properly filled out (ink, signed, etc)? YES...NO...NA

18. Did you sign the custody papers in the appropriate place? YES...NO...NA

19. Were correct containers used for the analysis requested? YES...NO...NA

20. Was sufficient amount of sample sent in each container? YES...NO...NA

I certify that I entered this project into LIMS and answered questions 17-20 (intial) _____

I certify that I attached a label with the unique LIMS number to each container (intial) _____

21. Were there Non-Conformance issues at login? YES...NO Was a PIPE generated? YES...NO...#_____

SECTION 25

ASSURING THE QUALITY OF TEST RESULTS
(NELAC 5.5.9)

25.1 OVERVIEW

In order to assure our clients of the validity of their data, the laboratory continuously evaluates the quality of the analytical process. The analytical process is controlled not only by instrument calibration as discussed in Section 21, but also by routine process quality control measurements (e. g., Blanks, Laboratory Control Samples (LCS), Matrix Spikes (MS), duplicates (DUP), surrogates, Internal Standards (IS)). These quality control checks are performed as required by the method or regulations to assess precision and accuracy. In addition to the routine process quality control samples, Proficiency Testing (PT) Samples (concentrations unknown to laboratory) are analyzed to help ensure laboratory performance.

25.2 CONTROLS

Sample preparation or pre-treatment is commonly required before analysis. Typical preparation steps include homogenization, grinding, solvent extraction, sonication, acid digestion, distillation, reflux, evaporation, drying and ashing. During these pre-treatment steps, samples are arranged into discreet manageable groups referred to as preparation (prep) batches. Prep batches provide a means to control variability in sample treatment. Control samples are added to each prep batch to monitor method performance and are processed through the entire analytical procedure with investigative/field samples.

25.3 NEGATIVE CONTROLS

25.3.1 **Method Blanks** are used to assess preparation and analysis for possible contamination during the preparation and processing steps.

25.3.1.1 The method blank is prepared from a clean matrix similar to that of the associated samples that is free from target analytes (e. g., Reagent water, Ottawa sand, glass beads, etc.) and is processed along with and under the same conditions as the associated samples.

25.3.1.2 The method blank goes through all of the steps of the process (including as necessary: filtration, clean-ups, etc.).

25.3.1.3 The specific frequency of use for method blanks during the analytical sequence is defined in the specific standard operating procedure for each analysis. Generally it is 1 for each batch of samples; not to exceed 20 environmental samples.

- Evaluation criteria and corrective action for method blanks is defined in the specific standard operating procedure for each analysis.

25.3.2 **Calibration Blanks** are prepared and analyzed along with calibration standards where applicable. They are prepared using the same reagents that are used to prepare the standards. In some analyses the calibration blank may be included in the calibration curve.

These blanks may be termed Initial Calibration Blanks (ICB) or Continuing Calibration Blanks (CCB).

25.3.3 **Instrument Blanks** are blank reagents or reagent water that may be processed during an analytical sequence in order to assess contamination in the analytical system. In general, instrument blanks are used to differentiate between contamination caused by the analytical system and that caused by the sample handling or sample prep process. Instrument blanks may also be inserted throughout the analytical sequence to minimize the effect of carryover from samples with high analyte content.

25.3.4 **Trip Blanks** are required to be submitted by the client with each shipment of samples requiring aqueous and solid volatiles analyses. Additionally, trip blanks may be prepared and analyzed for volatile analysis of air samples, when required by the client. A trip blank may be purchased (certified clean) or is prepared by the laboratory by filling a clean container with pure deionized water that has been purged to remove any volatile compounds. Appropriate preservatives are also added to the container. The trip blank is sent with the bottle order and is intended to reflect the environment that the containers are subjected to throughout shipping and handling and help identify possible sources if contamination is found. The field sampler returns the trip blank in the cooler with the field samples.

25.3.5 **Field Blanks** are sometimes used for specific projects by the field samplers. A field blank prepared in the field by filling a clean container with pure reagent water and appropriate preservative, if any, for the specific sampling activity being undertaken. (EPA OSWER)

25.3.6 **Equipment Blanks** are also sometimes created in the field for specific projects. An equipment blank is a sample of analyte-free media which has been used to rinse common sampling equipment to check effectiveness of decontamination procedures. (NELAC)

25.3.7 **Holding Blanks**, also referred to as refrigerator or freezer blanks, are used to monitor the sample storage units for volatile organic compounds during the storage of VOA samples in the laboratory. (Refer to section 24.4 and SOP Refrigerator and Freezer Holding Blanks for Volatile Sample Storage Units / NV08-224).

25.3.8 Field blanks, equipment blank and trip blanks, when received, are analyzed in the same manner as other field samples. Blanks are not knowingly selected for matrix QC, as it does not provide information on the behavior of the target compounds in the field samples. Usually, the client sample ID provides information to identify the different blanks with labels such as "FB", "EB", or "TB".

25.4 POSITIVE CONTROLS

Control samples (e. g., QC indicators) are analyzed with each batch of samples to evaluate data based upon (1) Method Performance (Laboratory Control Sample (LCS) or Blank Spike (BS)), which entails both the preparation and measurement steps; and (2) Matrix Effects (Matrix Spike (MS) (matrix spikes are not applicable to air) or Sample Duplicate (MD, DUP), which evaluate field sampling accuracy, precision, representativeness, interferences, and the effect of the matrix on the method performed. Each regulatory program and each method within those programs specify the control samples that are prepared and/or analyzed with a specific batch

Note that frequency of control samples vary with specific regulatory, methodology and project specific criteria. Complete details on method control samples are as listed in each analytical SOP.

25.4.1 Method Performance Control - Laboratory Control Sample (LCS)

25.4.1.1 The LCS measures the accuracy of the method in a blank matrix and assesses method performance independent of potential field sample matrix affects in a laboratory batch.

25.4.1.2 The LCS is prepared from a clean matrix similar to that of the associated samples that is free from target analytes (for example: Reagent water, Ottawa sand, glass beads, Teflon Chips, etc.) and is processed along with and under the same conditions as the associated samples. The LCS is spiked with verified known amounts of analytes or is made of a material containing known and verified amounts of analytes, taken through all preparation and analysis steps along with the field samples. Where there is no preparation taken for an analysis (such as in aqueous volatiles), or when all samples and standards undergo the same preparation and analysis process (such as Phosphorus), an initial calibration verification standard is reported as the LCS, as long as the more stringent criteria is met.

25.4.1.3 Certified pre-made reference material purchased from a NIST/A2LA accredited vendor may also be used for the LCS when the material represents the sample matrix or the analyte is not easily spiked (e. g., solid matrix LCS for metals, TDS, etc.).

25.4.1.4 The LCS is made from a different source from the calibration standard source.

25.4.1.5 As stated in the opening of this section, the LCS goes through all of the steps of the process (including as necessary: filtration, clean-ups, etc.).

25.4.1.6 The specific frequency of use for LCS during the analytical sequence is defined in the specific standard operating procedure for each analysis. It is generally 1 for each batch of samples; not to exceed 20 environmental samples.

25.4.1.7 If the mandated or requested test method, or project requirements, do not specify the spiking components, the laboratory spikes all reportable components to be reported in the Laboratory Control Sample (and Matrix Spike) where applicable (e.g. no spike of pH). However, in certain cases where the components interfere with accurate assessment (such as simultaneously spiking chlordane, toxaphene and PCBs in Method 608), the test method has an extremely long list of components or components are incompatible, at a minimum, a representative number of the listed components (see below) are used to control the test method. The selected components of each spiking mix must represent all chemistries, elution patterns and masses, permit specified analytes and other client requested components. However, the laboratory must also ensure that all reported components are used in the spike mixture within a two-year time period.

25.4.1.7.1 For methods that have 1-10 target analytes, spike all components.

25.4.1.7.2 For methods that include 11-20 target analytes, spike at least 10 or 80%, whichever is greater.

25.4.1.7.3 For methods with more than 20 target analytes, spike at least 16 components.

25.4.1.7.4 Exception: Due to analyte incompatibility in pesticides, toxaphene and chlordane are only spiked at client request based on specific project needs.

25.4.1.7.5 Exception: Due to analyte incompatibility between the various PCB aroclors, aroclors 1016 and 1260 are used for spiking as they cover the range of all of the aroclors. Specific aroclors may be used by request on a project specific basis.

25.4.1.8 **Accuracy Calculation**: Percent Recovery (%R) calculation (applies to LCS, CCV, Surrogates, and Matrix Spikes.

$$\%R = \frac{AV}{TV} \times 100$$

where: AV = Analyzed Value
TV = True Value

25.5 SAMPLE MATRIX CONTROLS

25.5.1 Matrix Spikes (MS)

25.5.1.1 The Matrix Spike is used to assess the effect that sample matrix of the spiked sample has on the precision and accuracy of the results generated by the method used.

25.5.1.2 An MS is essentially a sample fortified with a known amount of the test analyte(s). At a minimum, with each matrix-specific batch of samples processed, an MS is carried through the complete analytical procedure. Unless specified by the client, samples used for spiking are randomly selected and rotated between different client projects.

25.5.1.3 If the mandated or requested test method does not specify the spiking components, the laboratory spikes all reportable components to be reported in the Laboratory Control Sample and Matrix Spike. However, in certain cases where the components interfere with accurate assessment (such as, simultaneously spiking chlordane, toxaphene and PCBs in Method 608), the test method has an extremely long list of components or components are incompatible, a representative number of the listed components (see LCS analytes 25.4.1.6 above) are used to control the test method. The selected components of each spiking mix must represent all chemistries, elution patterns and masses, permit-specified analytes and other client requested components. However, the laboratory must also ensure that all reported components are used in the spike mixture within a two-year time period.

25.5.1.4 The percent recovery calculation for matrix spikes is essentially the same as the calculation shown in 25.4.1.8 except that:

AV = Sp – Sa

where: Sp = Spike result
Sa = Sample result

25.5.2 Surrogate Spikes

25.5.2.1 Surrogate Spikes are similar to matrix spikes except the analytes are compounds with properties that mimic the analyte of interest and are unlikely to be found in environment samples.

25.5.2.2 Surrogate compounds are added to all samples, standards, and blanks, for all organic chromatography methods except when the matrix precludes its use or when a surrogate is not available. The recovery of the surrogates is compared to the acceptance limits for the specific method (also refer to Section 25.5). Poor surrogate recovery may indicate a problem with sample composition and must be reported, with data qualifiers, to the client whose sample produced poor recovery. See specific method SOPs and Section 13 for the discussion of corrective actions.

25.5.3 Duplicates

25.5.3.1 For a measure of analytical precision, with each matrix-specific batch of samples processed, a matrix duplicate (MD or DUP) sample, matrix spike duplicate (MSD), or LCS duplicate (LCSD) is carried through the complete analytical procedure. Duplicate samples are usually analyzed with methods that do not require matrix spike analysis. LCSD's are normally not performed except when regulatory agencies or client specifications require them. The recoveries for the spiked duplicate samples must meet the same laboratory established recovery limits as the accuracy QC samples. If an LCSD is analyzed, both the LCS and LCSD must meet the same recovery criteria and be included in the final report. The precision measurement is reported as "Relative Percent Difference" (RPD). Poor precision between duplicates (except LCS/LCSD) may indicate non-homogeneous matrix or sampling.

25.5.3.2 Precision Calculation (Relative Percent Difference - RPD)

$$RPD = \frac{|S - D|}{\frac{(S + D)}{2}} \times 100$$

where: S=Sample Concentration
D=Duplicate Concentration

25.5.4 Internal Standards

25.5.4.1 When required in organic methods, internal standards are spiked into all environmental and quality control samples (including the initial calibration standards).

An internal standard is also used with some metals analyses. It is added to sample extracts after the extraction (post-prep). Consult the method-specific SOPs for details on the internal standard compounds, calculations and acceptance criteria.

25.6 ACCEPTANCE CRITERIA (CONTROL LIMITS)

25.6.1 As mandated by the test method and regulation, each individual analyte in the LCS, MS, or Surrogate Spike is evaluated against the control limits published in the test method. Where there are no established acceptance criteria, the laboratory calculates in-house control limits with the use of control charts or, in some cases, utilizes client project specific control limits or regulatory mandated control limits. When this occurs, the regulatory or project limits supersede the laboratory's in-house limits.

Note: For methods, analytes and matrices with very limited data (e.g., unusual matrices not analyzed often), interim limits are established using available data or by analogy to similar methods or matrices.

25.6.2 Once control limits have been established, they are verified, reviewed, and updated, if necessary, on an annual basis unless the method requires more frequent updating (e. g., EPA SW846 8000 series methods). Control limits are established per method (as opposed to per instrument) regardless of the number of instruments utilized.

25.6.2.1 The lab is to consider the effects of the spiking concentration control limits, and to avoid censoring of data. The acceptance criteria for recovery and precision are often a function of the spike concentration used. Therefore, caution must be used when pooling data to generate control limits.

25.6.2.2 Not only are the results to all be from a similar matrix, but the spiking levels are also approximately the same (within a factor of 2). Similarly, the matrix spike and surrogate results must all be generated using the same set of extraction, cleanup and analysis techniques. For example, results from solid samples extracted by ultrasonic extraction are not mixed with those extracted by Soxhlet.

25.6.2.3 The laboratory avoids discarding data that do not meet a preconceived notion of acceptable performance. This results in a censored data set, which, when used to develop acceptance criteria, leads to unrealistically narrow criteria. For a 99% confidence interval, 1 out of every 100 observations likely will still fall outside the limits. For methods with long analyte lists this may mean occasional failures every batch or two. While professional judgment is important in evaluating data to be used to develop acceptance criteria, specific results are not discarded simply because they do not meet one's expectations. However, data points are discarded if they were the result of human or mechanical error or sample concentration exceeded spike level by > 4x. (Right clicking on the control chart and selecting View Data from the drop down menu allows viewing a table of all the charted points with any qualifiers. This assists in determining if any points are to be discarded prior to limit generation.)

25.6.3 Laboratory generated % Recovery acceptance (control) limits are generally established by taking $\pm$ 3 Standard Deviations (99% confidence level) from the average

recovery of a minimum of 20-30 data points (more points are preferred). The system defaults to collecting the previous three months' data. This time frame is shortened if there are more than 200 points since the system slows down tremendously. The time frame is extended if there are not 20-30 points].

25.6.3.1 Regardless of the calculated limit, the limit is no tighter than the Calibration Verification (ICV/CCV) (unless the analytical method specifies a tighter limit).

25.6.3.2 The lowest acceptable recovery limit is 10% (the analyte must be detectable and identifiable). Exception: The lowest acceptable recovery limit for Benzidine will be 5% and the analyte must be detectable and identifiable.

25.6.3.3 The maximum acceptable recovery limit is 150%.

25.6.3.4 The maximum acceptable RPD limit is 50%. The minimum RPD limit is 10%.

25.6.3.5 If either the high or low end of the control limit changes by $\leq$ 5% from previous, the control chart is visually inspected and, using professional judgment, they may be left unchanged if there is no affect on laboratory ability to meet the existing limits.

25.6.3.6 The QA department generates a Control Limits Manual that contains tables summarizing the precision and accuracy acceptability limits for analyses performed at TestAmerica Nashville. The Control Limits Manual is considered to be part of the QA Manual and therefore carries the same revision number as the current QA Manual. Letter suffixes are added to the revision number if it becomes necessary to update the Control Limits Manual during the course of the year prior to a QA manual update. This manual includes an effective date, is updated each time new limits are generated and is located in each analytical department and the QA office. Unless otherwise noted, limits within these tables are laboratory generated. The analysts are instructed to use the current limits in the laboratory (dated and approved by the Technical Director and QA Manager) and entered into the LIMS. The Quality Assurance department maintains an archive of all limits used within the laboratory, as described in SOP Archiving / NV08-194. Archived limits are available upon request.

25.6.3.7 Client or contract required control limits are evaluated against the laboratory's statistically derived control limits to determine if the data quality objectives (DQOs) can be achieved. If laboratory control limits are not consistent with DQOs, then alternatives must be considered, such as method improvements or use of an alternate analytical method.

25.6.4 A LCS that is within the acceptance criteria establishes that the analytical system is in control and is used to validate the process. Samples that are analyzed with an LCS with recoveries outside of the acceptance limits may be determined as out of control and are reanalyzed if possible. If reanalysis is not possible, then the results for all affected analytes for samples within the same batch must be qualified when reported. The internal corrective action process (see Section 13) is also initiated if an LCS exceeds the acceptance limits. Sample results may be qualified and reported without reanalysis if the analyte results are below the

reporting limit and the LCS is above the upper control limit. See specific method SOPs for complete discussion of acceptance criteria and corrective action.

25.6.5 If the MS/MSDs do not meet acceptance limits, the MS/MSD and the associated spiked sample is reported with a qualifier for those analytes that do not meet limits. If obvious preparation errors are suspected, or if requested by the client, unacceptable MS/MSDs are reprocessed and reanalyzed to prove matrix interference. A more detailed discussion of acceptance criteria and corrective action can be found in the lab's method SOPs and in Section 13.

25.6.6 If a surrogate standard falls outside the acceptance limits, if there is not obvious chromatographic matrix interference, reanalyze the sample to confirm a possible matrix effect. If the recoveries confirm or there was obvious chromatographic interference, results are reported from the original analysis and a qualifier is added. Dilutions or cleanup procedures may be required when obvious chromatographic interference is present. If the reanalysis meets surrogate recovery criteria, the second run is reported (or both are reported if requested by the client). See specific method SOPs for a more detailed discussion of acceptance criteria and corrective action.

25.7 METHOD DETECTION LIMITS (MDLS)

MDLs, calculated as described in Section 20.7, are updated or verified annually, or more often if required by the method. Once values are approved, they are distributed to the analysts, entered in LIMS analyte by analyte, and tabulated in the Control Limits Manual.

25.8 ADDITIONAL PROCEDURES TO ASSURE QUALITY CONTROL

25.8.1 The laboratory has written and approved method SOPs procedures to assure the accuracy of the test method including calibration (see Section 21), use of certified reference materials (see Section 22) and use of PT samples (see Section 16).

25.8.2 A discussion regarding MDLs, Limit of Detection (LOD) and Limit of Quantitation (LOQ) can be found in Section 20.

25.8.3 Use of formulae to reduce data is discussed in the method standard operating procedures and in Section 21.

25.8.4 Selection of appropriate reagents and standards is included in Section 9 and 22.

25.8.5 A discussion on selectivity of the test is included in Section 5.

25.8.6 Constant and consistent test conditions are discussed in Section 19.

25.8.7 The laboratories sample acceptance policy is included in Section 24.

SECTION 26

REPORTING RESULTS
(NELAC 5.5.10)

26.1 OVERVIEW

The results of each test are reported accurately, clearly, unambiguously, and objectively in accordance with State and Federal regulations as well as client requirements. Analytical results are issued in a format that is intended to satisfy customer and laboratory accreditation requirements as well as provide the end user with the information needed to properly evaluate the results. Where there is conflict between client requests and laboratory ethics or regulatory requirements, the laboratory's ethical and legal requirements are paramount, and the laboratory must work with the client during project set up to develop an acceptable solution. A variety of report formats are available to meet specific needs.

In cases where a client asks for simplified reports, there must be a written request from the client. There still must be enough information that would show any analyses that were out of conformance (QC out of limits) and there must be a reference to a full report that is made available to the client.

Review of reported data is included in Section 20.

26.2 TEST REPORTS

Analytical results are reported in a format that is satisfactory to the client and meets all requirements of applicable accrediting authorities and agencies. A variety of report formats are available to meet specific needs. The report is printed on laboratory letterhead, reviewed, and signed by the appropriate project manager. At a minimum, the standard laboratory report must contain the following information:

26.2.1 A report title (e. g., Analytical Report for Samples) with a "sample results" column header.

26.2.2 Each report page printed on company letterhead which includes the laboratory name, address and telephone number.

26.2.3 A unique identification of the report (e. g., work order number) and on each page an identification in order to ensure the page is recognized as part of the report and a clear identification of the end.

Note: Page numbers of report are represented as page # of ##, where the first number is the page number and the second is the total number of pages. Pages are automatically paginated by LIMS when the report is created.

26.2.4 A copy of the chain of custody (COC).

- Any COCs involved with subcontracting are included.

- There is a statement on the front of the report that says, "The Chain of Custody, X page(s), is included and is an integral part of this report." The number of pages of the CoC (X) is calculated automatically by the number of pages that are scanned in the CoC folder. Any pages scanned with the CoC (i. e., non-conformances, emails, etc.) are included in the page count.

- Any additional addenda to the report must be treated in a similar fashion, so it is a recognizable part of the report and cannot accidentally get separated from the report (e. g., sampling information).

26.2.5 The name and address of client and a project name/number, if applicable.

26.2.6 Client project manager or other contact

26.2.7 Description and unambiguous identification of the tested sample(s) including the client identification code.

26.2.8 Date of receipt of sample, date and time of collection, and date(s) of test preparation and performance, and time of preparation or analysis if the required holding time for either activity is less than or equal to 72 hours. The analysis time is always included on the report. The preparation date and time are included for those methods that require a separate preparation.

26.2.9 Date reported or date of revision, if applicable.

26.2.10 Method of analysis including method code (EPA, Standard Methods, etc).

26.2.11 Reporting limit.

26.2.12 Method detection limits (if requested)

26.2.13 Definition of data qualifiers and reporting acronyms (e. g., ND).

26.2.14 Sample results.

26.2.15 QC data consisting of method blank, surrogate, LCS, and MS/MSD recoveries and control limits.

26.2.16 Condition of samples at receipt including temperature (included on the Cooler Receipt Form, which is part of the CoC).

26.2.17 A statement to the effect that the results relate only to the items tested and the sample as received by the laboratory.

26.2.18 A statement that the report shall not be reproduced except in full, without prior express written approval by the laboratory.

26.2.19 A signature and title of the person(s) accepting responsibility for the content of the report and date of issue. Signatories are appointed by the Lab Director. For applying an

electronic signature, refer to the Electronic Signature Policy (Section 26.4) and the Corporate SOP on Electronic Reporting and Signature Policy (No. CA-I-P-002).

26.2.20 When NELAC accreditation is required, the lab certifies that the test results meet all requirements of NELAC or provide reasons and/or justification if they do not. Accreditations/certifications are included near the end of the report, listed by method.

26.2.21 The laboratory includes a cover letter.

26.2.22 Where applicable, a narrative to the report that explains the issue(s) and corrective action(s) taken in the event that a specific accreditation or certification requirement was not met.

26.2.23 When soil samples are analyzed, a specific identification as to whether soils are reported on a "wet weight" or "dry weight" basis.

26.2.24 Appropriate laboratory certification number for the state of origin of the sample, if applicable.

26.2.25 If only part of the report is provided to the client (client requests some results before all of it is complete), it must be clearly indicated on the report (e. g., partial report, draft report). A complete report must be sent once all of the work has been completed.

26.2.26 Any non-TestAmerica subcontracted analysis results are provided as a separate report on the official letterhead of the subcontractor. All TestAmerica network subcontracting is clearly identified on the report as to which laboratory performed a specific analysis.

26.2.27 Reports for Ohio VAP work require a VAP affidavit be completed and included with the report.

26.3 REPORTING LEVEL OR REPORT TYPE

TestAmerica Nashville offers three levels of quality control reporting. Each level, in addition to its own specific requirements, contains all the information provided in the preceding level. The packages provide the following information in addition to the information described above:

- Level II is a report plus summary information, including results for the method blank reported to the laboratory MDL, percent recovery for laboratory control samples and matrix spike samples, and the RPD values for all MSD and sample duplicate analyses.
- Level III contains all the information supplied in Level II, but presented on the CLP-like summary forms, and relevant calibration information. A Level II report is not included, unless specifically requested. No raw data is provided.
- Level IV is the same as Level III with the addition of all raw supporting data.

In addition to the various levels of QC packaging, the laboratory also provides reports in diskette deliverable form. Procedures used to ensure client confidentiality are outlined in Section 26.7.

26.3.1 Electronic Data Deliverables (EDDs)

EDDs are routinely offered as part of TestAmerica's services. TestAmerica Nashville offers a variety of EDD formats including Environmental Restoration Information Management System (ERPIMS), EXCEL, dBASE, GISKEY, and Text Files.

EDD specifications are submitted to the IT department by the PM for review and undergo the contract review process. Once the facility has committed to providing data in a specific electronic format, the coding of the format may need to be performed. This coding is documented and validated. The validation of the code is retained by the IT staff coding the EDD.

EDDs are subject to a review to ensure their accuracy and completeness. If EDD generation is automated, review may be reduced to periodic screening if the laboratory can demonstrate that it can routinely generate that EDD without errors. Any revisions to the EDD format must be reviewed until it is demonstrated that it can routinely be generated without errors. If the EDD can be reproduced accurately and if all subsequent EDDs can be produced error-free, each EDD does not necessarily require a review.

26.4 ELECTRONIC REPORTING AND SIGNATURE POLICY

Following the lead of the Federal Paperwork Reduction Act, TestAmerica has implemented policies and procedures to help reduce paper usage. One of these procedures is to generate final reports and provide them to clients in pdf format.

Laboratory Director appointed representatives may approve final reports using an electronic signature that is applied to the report at the time of generation. This policy is prepared to state that the electronically applied signatures on TestAmerica Nashville reports are as legally binding as a handwritten "wet signature." This policy is intended to prevent the possibility of non-repudiation (denial that an individual signed the document) and to insure authenticity and security. In order to insure the electronic signatures are valid and unequivocally represent the identity of the signer, TestAmerica uses 21 CFR Part 11 "Electronic Records; Electronic Signatures" from the FDA as well as EPA's procurement policy (EPS 00-01) as guidance documents for this policy.

In order to ensure authenticity of the reports, the following conditions must be met:

26.4.1 Report Content

- State that the report was electronically signed.
- The printed name and title of the signer must be underneath the signature.
- The date and time when the signature was executed is represented in the "Report Issued" entry on the cover page of the report.
- The meaning of the signature: (e. g., reviewed and approved).

In order to insure the integrity of the signatures, the following security features have been implemented.

26.4.2 General Requirements

- The identity of the signatory must be verified before an electronic signature can be created for that person.
- Each electronic signature must be unique to a single individual and must not be reused by or assigned to another individual
- Persons using an electronic signature must certify that the electronic signatures in the system are intended to be the legally binding equivalent to their traditional handwritten signature. On this certification, the signatory states that their passwords are to remain completely confidential and can only be used by the genuine owner of the password and the sign-off does not take place until each page has been viewed.

26.4.3 Components and Controls

Two distinct identification components are utilized for each individual. The components are a) user name b) password. Each signing requires that the entry of the username and the password be reentered. The signatures are not copied, excised or transferred from the report by ordinary means.

The report is not changed once the signature has been applied and the pdf files are stored on the file server with security as well as password protected to ensure no changes are made to the file.

"pdf" reports must be backed up on a magnetic tape or other durable storage media (e. g., DVD) and maintained secure for up to 10 years.

26.5 SUPPLEMENTAL INFORMATION FOR TEST

The lab identifies any unacceptable QC analyses or any other unusual circumstances or observations such as environmental conditions and any non-standard conditions that may have affected the quality of a result. This is typically in the form of a footnote or a qualifier and/or a narrative explaining the discrepancy in the front of the report. Refer to Appendix 7 for a list of the laboratory's standard footnotes and qualifiers.

26.5.1 Numeric results with values outside of the calibration range, either high or low are qualified as "estimated."

26.5.2 Where quality system requirements are not met, a statement of compliance/non-compliance with requirements and/or specifications, including identification of test results derived from any sample that did not meet NELAC sample acceptance requirements such as improper container, holding time, or temperature.

26.5.3 Where applicable, a statement on the estimated uncertainty of measurements; information on uncertainty is needed when a client's instructions so require.

26.5.4 Opinions and Interpretations - The test report contains objective information, and generally does not contain subjective information such as opinions and interpretations. If such information is required by the client, the Laboratory Director must determine if a response can be prepared. If so, the Laboratory Director designates the appropriate member of the management team to prepare a response. The response must be fully documented, and reviewed by the Laboratory Director, before release to the client. There may be additional fees charged to the client at this time, as this is a non-routine function of the laboratory.

When opinions or interpretations are included in the report, the laboratory provides an explanation as to the basis upon which the opinions and interpretations have been made. Opinions and interpretations are clearly noted as such and where applicable, and a comment must be added suggesting that the client verify the opinion or interpretation with their regulator.

26.6 ENVIRONMENTAL TESTING OBTAINED FROM SUBCONTRACTORS

If TestAmerica Nashville is not able to provide the client the requested analysis, the samples are subcontracted following the procedures outlined in Section 8.

Data reported from analyses performed by a subcontractor laboratory are clearly identified as such on the analytical report provided to the client. Results from a subcontract laboratory outside of the TestAmerica network are reported to the client on the subcontract laboratory's original report stationary and the report includes any accompanying documentation.

26.7 CLIENT CONFIDENTIALITY

In situations involving the transmission of environmental test results by telephone, facsimile or other electronic means, client confidentiality must be maintained.

TestAmerica must not intentionally divulge to any person (other than the Client or any other person designated by the Client in writing) any information regarding the services provided by TestAmerica or any information disclosed to TestAmerica by the Client. Furthermore, information known to be potentially endangering to national security or an entity's proprietary rights must not be released.

Note: This does not apply to the extent that the information is required to be disclosed by TestAmerica under the compulsion of legal process. TestAmerica does, to the extent feasible, provide reasonable notice to the client before disclosing the information.

Note: Authorized representatives of an accrediting authority are permitted to make copies of any analyses or records relevant to the accreditation process, and copies may be removed from the laboratory for purposes of assessment.

26.7.1 Report deliverable formats are discussed with each new client. If a client requests that reports be faxed or e-mailed, the reports are faxed with a cover sheet or e-mailed with the following note that includes a confidentiality statement similar to the following:

This material is intended only for the use of the individual(s) or entity to whom it is addressed, and may contain information that is privileged and confidential. If you are not the intended

recipient, or the employee or agent responsible for delivering this material to the intended recipient, you are hereby notified that any dissemination, distribution or copying of this communication is strictly prohibited. If you have received this communication in error, please notify us immediately by telephone at the 1-800-765-0980 (or for e-mails: please notify us immediately by e-mail or by phone (1-800-765-0980) and delete this material from any computer).

26.8 FORMAT OF REPORTS

The format of reports is designed to accommodate each type of environmental test carried out and to minimize the possibility of misunderstanding or misuse.

26.9 AMENDMENTS TO TEST REPORTS

Corrections, additions, or deletions to reports are only made when justification arises through supplemental documentation. Justification is documented using the laboratory's corrective action system (refer to Section 13).

The revised report is retained on the Archive data server, as is the original report. When the pdf of a report is created, the pdf name has the work order number and the date and time that the pdf was created. The original report has the earliest date, and any revisions have later dates and times.

When the report is re-issued, a notation that the report has been revised is placed on the cover/signature page of the report along with a brief explanation of reason for the re-issue and a reference back to the last final report generated. For Example: *Report was revised on 11/3/07 to include toluene in sample NQA1504 per client's request. This final report replaces the final report generated on 10/27/07 at 10:47am.*

26.10 POLICIES ON CLIENT REQUESTS FOR AMENDMENTS

26.10.1 Sample Reanalysis Policy

Because there is a certain level of uncertainty with any analytical measurement a sample reanalysis may result in either a higher or lower value from an initial sample analysis. There are also variables that may be present (e.g. sample homogeneity, analyte precipitation over time, etc.) that may affect the results of a reanalysis. Based on the above comments, the laboratory reanalyzes samples at a client's request with the following caveats.

- Homogenous samples: If a reanalysis agrees with the original result to within the RPD limits for MS/MSD or Duplicate analyses, or within ±1 reporting limit for samples ≤ 5x the reporting limit, the original analysis is reported. At the client's request, both results may be reported on the same report but not on two separate reports.

- If the reanalysis does not agree (as defined above) with the original result, then the laboratory investigates the discrepancy and reanalyzes the sample a third time for confirmation if sufficient sample is available.

- Any potential charges related to reanalysis are discussed in the contract terms and conditions or discussed at the time of the request. The client is typically charged for reanalysis unless it is determined that the lab was in error.

- Due to the potential for increased variability, reanalysis may not be applicable to non-homogenous, Encore, and Sodium Bisulfate preserved samples. See the Department Supervisor, Technical Director, QA Manager, or Laboratory Director if unsure.

26.10.2 Policy on Data Omissions or Reporting Limit Increases

Fundamentally, our policy is simply to not omit previously reported results (including data qualifiers) or to not raise reporting limits and report sample results as ND. This policy has few exceptions. Exceptions are:

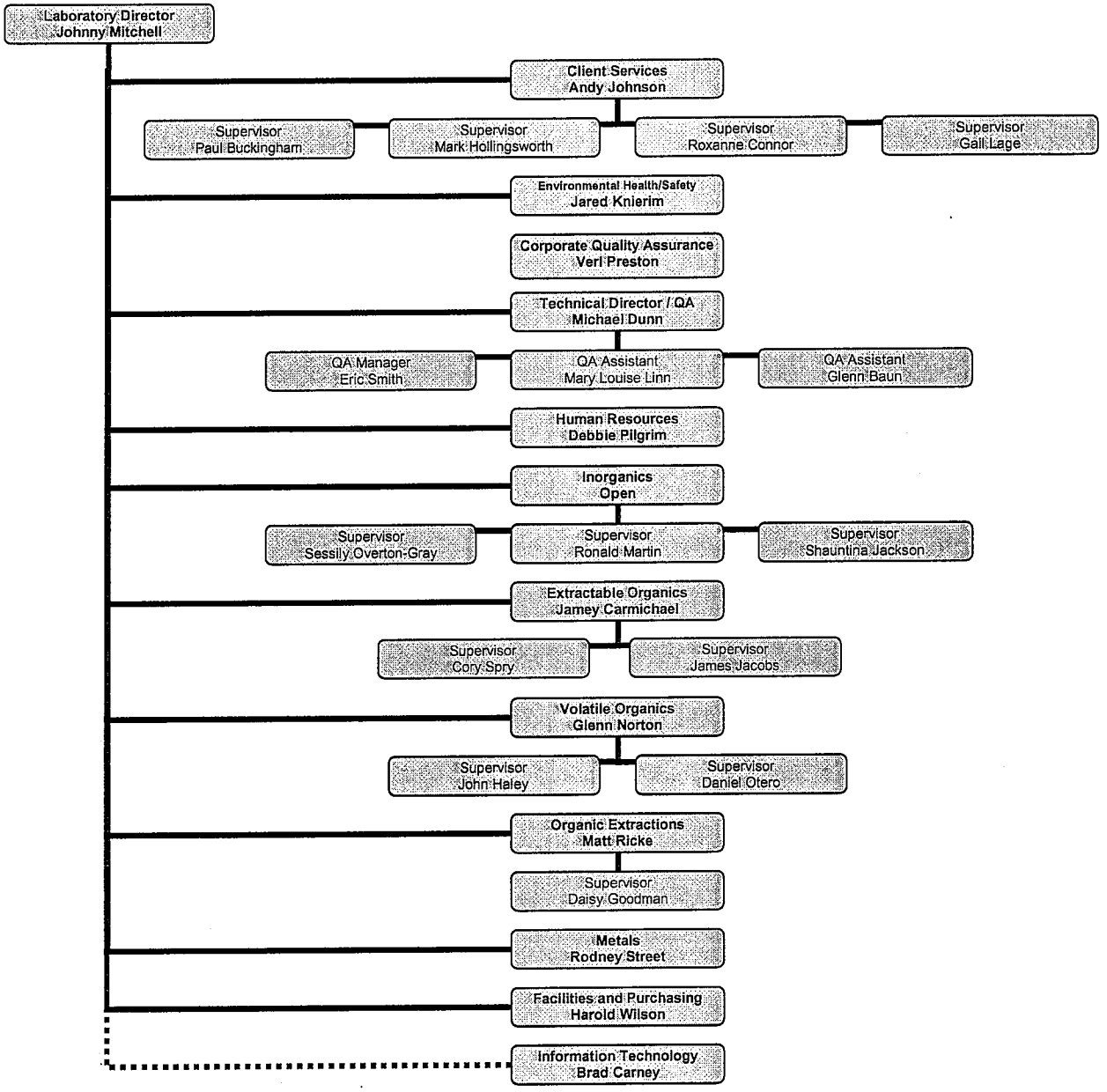
- Laboratory error.
- Sample identification is indeterminate (confusion between COC and sample labels).
- An incorrect analysis (not analyte) was requested (e. g., COC lists 8315 but client wanted 8310). A written request for the change is required.
- Incorrect limits reported based on regulatory requirements.
- The requested change has absolutely *no possible* impact on the interpretation of the analytical results and there is *no possibility* of the change being interpreted as misrepresentation by anyone inside or outside of our company.

26.10.3 Multiple Reports

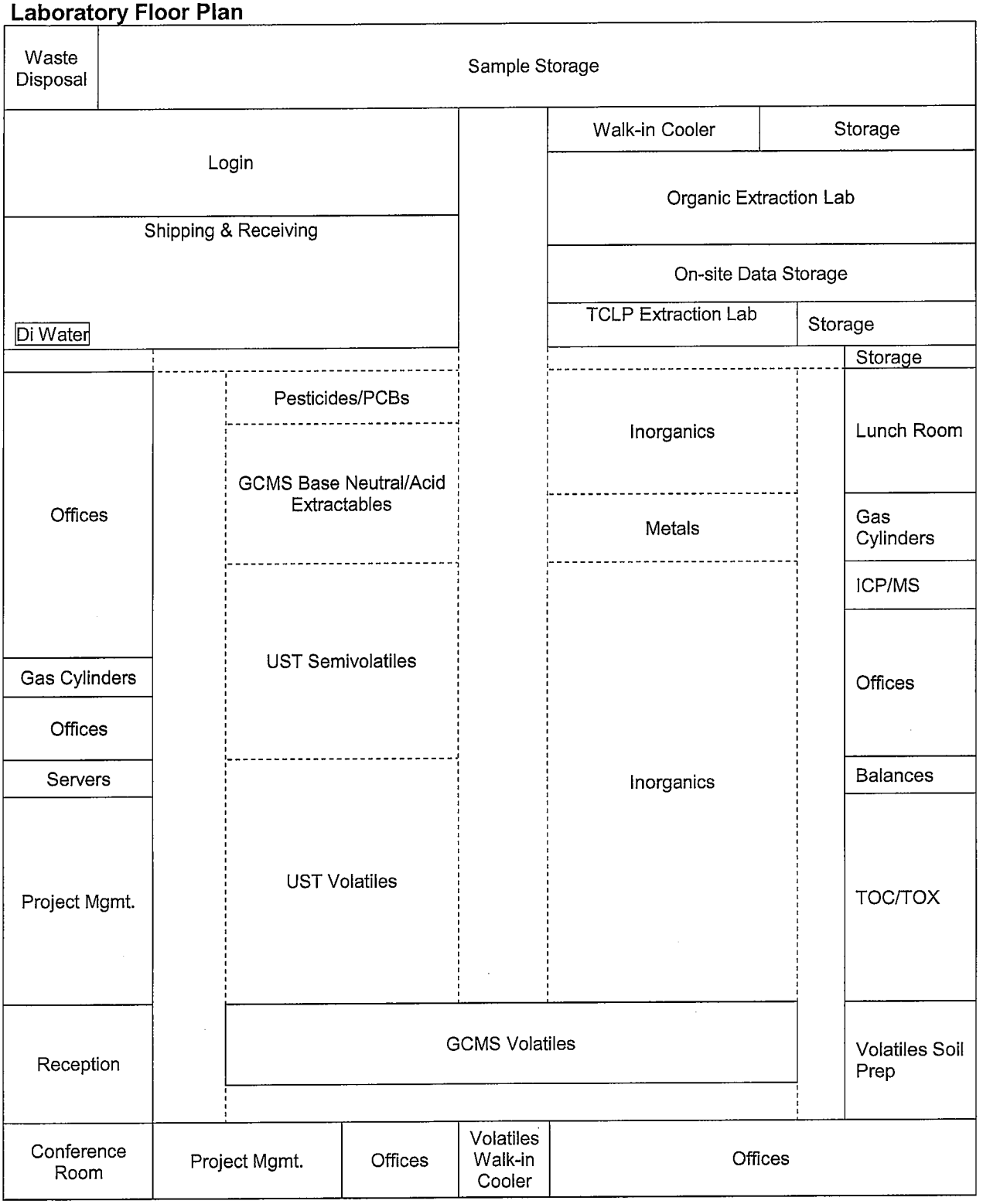
TestAmerica does not issue multiple reports for the same workorder where there is different information on each report (this does not refer to copies of the same report) unless required to meet regulatory needs and approved by the lab QA Manager.

(Reserved)

Laboratory Organization Chart



Laboratory Floor Plan



(Reserved)

Glossary/Acronyms

Glossary:

Acceptance Criteria:
Specified limits placed on characteristics of an item, process, or service defined in requirement documents. (ASQC)

Accreditation:

The process by which an agency or organization evaluates and recognizes a laboratory as meeting certain predetermined qualifications or standards, thereby accrediting the laboratory. In the context of the National Environmental Laboratory Accreditation Program (NELAP), this process is a voluntary one. (NELAC)

Accrediting Authority:
The Territorial, State, or Federal Agency having responsibility and accountability for environmental laboratory accreditation and which grants accreditation (NELAC) [1.5.2.3]

Accuracy:
The degree of agreement between an observed value and an accepted reference value. Accuracy includes a combination of random error (precision) and systematic error (bias) components which are due to sampling and analytical operations; a data quality indicator. (QAMS)

Aliquot
A measured portion of a sample used for analysis.

Analyst:
The designated individual who performs the "hands-on" analytical methods and associated techniques and who is the one responsible for applying required laboratory practices and other pertinent quality controls to meet the required level of quality. (NELAC)

Batch:
Environmental samples which are prepared and/or analyzed together with the same process and personnel, using the same lot(s) of reagents. A preparation batch is composed of one to 20 environmental samples of the same matrix, meeting the above mentioned criteria and with a maximum time between the start of processing of the first and last sample in the batch to be 24 hours. An analytical batch is composed of prepared environmental samples (extracts, digestates or concentrates) and /or those samples not requiring preparation, which are analyzed together as a group using the same calibration curve or factor. An analytical batch can include samples originating from various environmental matrices and can exceed 20 samples. (NELAC Quality Systems Committee)

Blank:
A sample that has not been exposed to the analyzed sample stream in order to monitor contamination during sampling, transport, storage or analysis. The blank is subjected to the usual analytical and measurement process to establish a zero baseline or background value and is sometimes used to adjust or correct routine analytical results. (ASQC)

Blind Sample:
A sample for analysis with a composition known to the submitter. The analyst/laboratory may know the identity of the sample but not its composition. It is used to test the analyst's or laboratory's proficiency in the execution of the measurement process.

Calibration:
To determine, by measurement or comparison with a standard, the correct value of each scale reading on a meter, instrument, or other device. The levels of the applied calibration standard must bracket the range of planned or expected sample measurements. (NELAC)

Calibration Curve:
The graphical relationship between the known values, such as concentrations, of a series of calibration standards and their instrument response. (NELAC)

Calibration Method:
A defined technical procedure for performing a calibration. (NELAC)

Calibration Standard:
A substance or reference material used to calibrate an instrument (QAMS)

Certified Reference Material (CRM):
A reference material one or more of whose property values are certified by a technically valid procedure, accompanied by or traceable to a certificate or other documentation which is issued by a certifying body. (ISO Guide 30–2.2)

Chain of Custody:
An unbroken trail of accountability that ensures the physical security of samples and includes the signatures of all who handle the samples. (NELAC) [5.12.4]

Clean Air Act:
The enabling legislation in 42 U>S>C> 7401 et seq., Public Law 91-604, 84 Stat. 1676 Pub. L. 95-95, 91 Stat., 685 and Pub. L. 95-190, 91 Stat., 1399, as amended, empowering EPA to promulgate air quality standards, monitor and enforce them. (NELAC)

Comprehensive Environmental Response, Compensation and Liability Act (CERCLA/SUPERFUND):
The enabling legislation in 42 U.S.C. 9601-9675 et seq., as amended by the Superfund Amendments and Reauthorization Act of 1986 (SARA), 42 U.S.C. 9601 et seq., to eliminate the health and environmental threats posed by hazardous waste sites. (NELAC)

Compromised Samples:
Those samples which are improperly sampled, insufficiently documented (chain of custody and other sample records and/or labels), improperly preserved, collected in improper containers, or exceeding holding times when delivered to a laboratory. Under normal conditions, compromised samples are not analyzed. If emergency situation require analysis, the results must be appropriately qualified. (NELAC)

Confidential Business Information (CBI):
Information that an organization designates as having the potential of providing a competitor with inappropriate insight into its management, operation or products. NELAC and its

representatives agree to safeguarding identified CBI and to maintain all information identified as such in full confidentiality.

Confirmation:
Verification of the identity of a component through the use of an approach with a different scientific principle from the original method. These may include, but are not limited to:

Second column confirmation
Alternate wavelength
Derivatization
Mass spectral interpretation
Alternative detectors or
Additional Cleanup procedures
(NELAC)

Conformance:
An affirmative indication or judgement that a product or service has met the requirements of the relevant specifications, contract, or regulation; also the state of meeting the requirements. (ANSI/ASQC E4-1994)

Continuing Calibration Blank:
solvent blank following CCV.

Continuing Calibration Verification: A standard from the calibration curve that is used to verify that the instrument remains properly calibrated throughout the analytical run.

Corrective Action:
The action taken to eliminate the causes of an existing nonconformity, defect or other undesirable situation in order to prevent recurrence. (ISO 8402)

Correction: Actions necessary to correct or repair analysis specific non-conformances. The acceptance criteria for method specific QC and protocols as well as the associated corrective actions. The analyst is most frequently the one to identify the need for this action as a result of calibration checks and QC sample analysis. No significant action is taken to change behavior, process or procedure.

Data Audit:
A qualitative and quantitative evaluation of the documentation and procedures associated with environmental measurements to verify that the resulting data re of acceptable quality (i.e., that they meet specified acceptance criteria). (NELAC)

Data Reduction:
The process of transforming raw data by arithmetic or statistical calculations, standard curves, concentration factors, etc., and collation into a more useable form. (EPA-QAD)

Deficiency:
An unauthorized deviation from acceptable procedures or practices, or a defect in an item. (ASQC)

Detection Limit:
The lowest concentration or amount of the target analyte that can be identified, measured, and reported with confidence that the analyte concentration is not a false positive value. See Method Detection Limit. (NELAC)

Document Control:
The act of ensuring that documents (and revisions thereto) are proposed, reviewed for accuracy, approved for release by authorized personnel, distributed properly, and controlled to ensure use of the correct version at the location where the prescribed activity if performed. (ASQC)

Duplicate Analyses:
The analyses or measurements of the variable of interest performed identically on two subsamples of the same sample. The results from duplicate analyses are used to evaluate analytical or measurement precision but not the precision of sampling, preservation or storage internal to the laboratory. (EPA-QAD)

Equipment Blank:
Sample of analyte-free media which has been used to rinse common sampling equipment to check effectiveness of decontamination procedures. (NELAC)

External Standard Calibration:
Calibrations for methods that do not utilize internal standards to compensate for changes in instrument conditions.

Federal Water Pollution Control Act (Clean Water Act, CWA):
The enabling legislation under 33 U.S.C. 1251 et seq., Public Law 92-50086 Stat 816, that empowers EPA to set discharge limitations, write discharge permits, monitor, and bring enforcement action for non-compliance. (NELAC)

Field Blank:
Blank prepared in the field by filing a clean container with pure de-ionized water and appropriate preservative, if any, for the specific sampling activity being undertaken (EPA OSWER)

Field of Testing:
NELAC's approach to accrediting laboratories by program, method and analyte. Laboratories requesting accreditation for a program-method-analyte combination or for an up-dated/improved method are required to submit to only that portion of the accreditation process not previously addressed (see NELAC, section 1.9ff). (NELAC)

Finding:
An assessment conclusion that identifies a condition having a significant effect on an item or activity. As assessment finding is normally a deficiency and is normally accompanied by specific examples of the observed condition. (NELAC)

Holding Times (Maximum Allowable Holding Times):
The maximum times that samples may be held prior to analyses and still be considered valid or not compromised. (40 CFR Part 136)

Initial Calibration Blank:
Solvent blank immediately following ICV.

Initial Calibration Verification:
A standard from a second source that is analyzed to verify the calibration curve.

Internal Standard:
A known amount of standard added to a test portion of a sample and carried through the entire measurement process as a reference for evaluating and controlling the precision and bias of the applied analytical test method. (NELAC)

Internal Standard Calibration:
Calibrations for methods that utilize internal standards to compensate for changes in instrument conditions.

Instrument Blank:
A clean sample (e.g., distilled water) processed through the instrumental steps of the measurement process; used to determine instrument contamination. (EPA-QAD)

Instrument Response:
Instrument response is normally expressed as either peak area or peak height however it may also reflect a numerical representation of some type of count on a detector (e.g. Photomultiplier tube, or Diode array detector) and is used in this document to represent all types.

Laboratory:
A defined facility performing environmental analyses in a controlled and scientific manner. (NELAC)

Laboratory Control Sample (however named, such as laboratory fortified blank, spiked blank, or QC check sample):
A sample matrix, free from the analytes of interest, spiked with verified known amounts of analytes or a material containing known and verified amounts of analytes, taken through all preparation and analysis steps. It is generally used to establish intra-laboratory or analyst specific precision and bias or to assess the performance of all or a portion of the measurement system.

An LCS must be prepared at a minimum of 1 per batch of 20 or less samples per matrix type per sample extraction or preparation method except for analytes for which spiking solutions are not available such as pH, color, odor, temperature, dissolved oxygen or turbidity. The results of these samples are used to determine batch acceptance.

Note: NELAC standards allow a matrix spike to be used in place of this control as long as the acceptance criteria are as stringent as for the LCS. (NELAC)

Laboratory Duplicate:
Aliquots of a sample taken from the same container under laboratory conditions and processed and analyzed independently. (NELAC)

Least Squares Regression (1st Order Curve):
The least squares regression is a mathematical calculation of a straight line over two axes. The y axis represents the instrument response (or Response ratio) of a standard or sample and the x axis represents the concentration. The regression calculation generates a correlation coefficient (r) that is a measure of the "goodness of fit" of the regression line to the data. A value

of 1.00 indicates a perfect fit. In order to be used for quantitative purposes, r must be greater than or equal to 0.99 for organics and 0.995 for inorganics.

Limit of Detection (LOD):
An estimate of the minimum amount of a substance that an analytical process can reliably detect. An LOD is analyte- and matrix-specific and may be laboratory dependent. (Analytical Chemistry, 55, p.2217, December 1983, modified) See also Method Detection Limit.

Matrix:
The component or substrate that contains the analyte of interest. For purposes of batch and QC requirement determinations, the following matrix distinctions ~~shall be~~ are to be used:

Aqueous: Any aqueous sample excluded from the definition of Drinking Water matrix or Saline/Estuarine source. Includes surface water, groundwater, effluents, and TCLP or other extracts.

Drinking Water: any aqueous sample that has been designated as a potable or potential potable water source.

Saline/Estuarine: any aqueous sample from an ocean or estuary, or other salt water source such as the Great Salt Lake.

Non-aqueous Liquid: any organic liquid with <15% settleable solids.

Biological Tissue: any sample of a biological origin such as fish tissue, shellfish, or plant material. Such samples must be grouped according to origin.

Solids: includes soils, sediments, sludges, and other matrices with >15% settleable solids.

Chemical Waste: a product or by-product of an industrial process that results in a matrix not previously defined.

Air: whole gas or vapor samples including those contained in flexible or rigid wall containers and the extracted concentrated analytes of interest from a gas or vapor that are collected with a sorbant tube, impinger solution, filter, or other device. (NELAC)

Matrix Spike (spiked sample or fortified sample):

Prepared by adding a known mass of target analyte to a specified amount of matrix sample for which an independent estimate of target analyte concentration is available. Matrix spikes are used, for example, to determine the effect of the matrix on a method's recovery efficiency.

Matrix spikes are performed at a frequency of one in 20 samples per matrix type per sample extraction or preparation method except for analytes for which spiking solutions are not available such as, total suspended solids, total dissolved solids, total volatile solids, total solids, pH, color, odor, temperature, dissolved oxygen or turbidity. The selected sample(s) are to be rotated among client samples so that various matrix problems may be noted and/or addressed. Poor performance in a matrix spike may indicate a problem with the sample composition and is reported to the client whose sample was used for the spike. (QAMS)

Matrix Spike Duplicate (spiked sample or fortified sample duplicate):
A second replicate matrix spike is prepared in the laboratory and analyzed to obtain a measure of the precision of the recovery for each analyte.

Matrix spike duplicates or laboratory duplicates are analyzed at a minimum of 1 in 20 samples per matrix type per sample extraction or preparation method. The laboratory documents their procedure to select the use of an appropriate type of duplicate. The selected sample(s) are to be rotated among client samples so that various matrix problems may be noted and/or addressed. Poor performance in the duplicates may indicate a problem with the sample composition and are reported to the client whose sample was used for the duplicate. (QAMS)

Method Blank:
A sample of a matrix similar to the batch of associated samples (when available) that is free from the analytes of interest and is processed simultaneously with and under the same conditions as samples through all steps of the analytical procedures, and in which no target analytes or interferences are present at concentrations that impact the analytical results for sample analyses. (NELAC)

Method Detection Limit:
The minimum concentration of a substance (an analyte) that can be measured and reported with 99% confidence that the analyte concentration is greater than zero and is determined from analysis of a sample in a given matrix containing the analyte. (40 CFR Part 136, Appendix B)

Method Reporting Limit:
The laboratory nominal Quantitation Limit (QL) or the level of sensitivity required by the client but not lower than the LOD.

Negative Control:
Measures taken to ensure that a test, its components, or the environment do not cause undesired effects, or produce incorrect test results. (NELAC)

Performance Audit:
The routine comparison of independently obtained qualitative and quantitative measurement system data with routinely obtained data in order to evaluate the proficiency of an analyst or laboratory. (NELAC)

Performance Based Measurement System (PBMS):
A set of processes wherein the data quality needs, mandates or limitations of a program or project are specified and serve as criteria for selecting appropriate test methods to meet those needs in a cost-effective manner. (NELAC)

Positive Control:
Measures taken to ensure that a test and/or its components are working properly and producing correct or expected results from positive test subjects. (NELAC)

Precision:
The degree to which a set of observations or measurements of the same property, obtained under similar conditions, conform to themselves; a data quality indicator. Precision is usually expressed as standard deviation, variance or range, in either absolute or relative terms. (NELAC)

Preservation:
Refrigeration and/or reagents added at the time of sample collection (or later) to maintain the chemical and/or biological integrity of the sample. (NELAC)

Proficiency Testing:
A means of evaluating a laboratory's performance under controlled conditions relative to a given set of criteria through analysis of unknown samples provided by an external source. (NELAC) [2.1]

Proficiency Testing Program:
The aggregate of providing rigorously controlled and standardized environmental samples to a laboratory for analysis, reporting of results, statistical evaluation of the results and the collective demographics and results summary of all participating laboratories. (NELAC)

Proficiency Test Sample (PT):
A sample, the composition of which is unknown to the analyst and is provided to test whether the analyst/laboratory can produce analytical results within specified acceptance criteria. (QAMS)

Quality Assurance:
An integrated system of activities involving planning, quality control, quality assessment, reporting and quality improvement to ensure that a product or service meets defined standards of quality with a stated level of confidence. (QAMS)

Quality Assurance [Project] Plan (QAPP):
A formal document describing the detailed quality control procedures by which the quality requirements defined for the data and decisions pertaining to a specific project are to be achieved. (EAP-QAD)

Quality Control:
The overall system of technical activities which purpose is to measure and control the quality of a product or service so that it meets the needs of users. (QAMS)

Quality Control Sample:
An uncontaminated sample matrix spiked with known amounts of analytes from a source independent from the calibration standards. It is generally used to establish intra-laboratory or analyst specific precision and bias or to assess the performance of all or a portion of the measurement system. (EPA-QAD)

Quality Manual:
A document stating the management policies, objectives, principles, organizational structure and authority, responsibilities, accountability, and implementation of an agency, organization, or laboratory, to ensure the quality of its product and the utility of its product to its users. (NELAC)

Quality System:
A structured and documented management system describing the policies, objectives, principles, organizational authority, responsibilities, accountability, and implementation plan of an organization for ensuring quality in its work processes, products (items), and services. The quality system provides the framework for planning, implementing, and assessing work performed by the organization and for carrying out required QA and QC (ANSI/ASQC-E-41994)

Quantitation Limits:
The maximum or minimum levels, concentrations, or quantities of a target variable (e.g., target analyte) that can be quantified with the confidence level required by the data user. (NELAC)

Range:
The difference between the minimum and the maximum of a set of values. (EPA-QAD)

Reagent Blank (method reagent blank):
A sample consisting of reagent(s), without the target analyte or sample matrix, introduced into the analytical procedure at the appropriate point and carried through all subsequent steps to determine the contribution of the reagents and of the involved analytical steps. (QAMS)

Reference Material:
A material or substance one or more properties of which are sufficiently well established to be used for the calibration of an apparatus, the assessment of a measurement method, or for assigning values to materials. (ISO Guide 30-2.1)

Reference Method:
A method of known and documented accuracy and precision issued by an organization recognized as competent to do so. (NELAC)

Reference Standard:
A standard, generally of the highest metrological quality available at a given location, from which measurements made at that location are derived. (VIM-6.0-8)

Replicate Analyses:
The measurements of the variable of interest performed identically on two or more sub-samples of the same sample within a short time interval. (NELAC)

Report Limit (RL):
The laboratory nominal Quantitation Limit (QL) or the level of sensitivity required by the client but not lower than the LOD.

Requirement:
Denotes a mandatory specification; often designated by the term "shall". (NELAC)

Resource Conservation and Recovery Act (RCRA):
The enabling legislation under 42 USC 321 et seq. (1976), that gives EPA the authority to control hazardous waste from the "cradle-to-grave", including its generation, transportation, treatment, storage, and disposal. (NELAC)

Safe Drinking Water Act (SDWA):
The enabling legislation, 42 USC 300f et seq. (1974), (Public Law 93-523), that requires the EPA to protect the quality of drinking water in the U.S. by setting maximum allowable contaminant levels, monitoring, and enforcing violations. (NELAC)

Sample Duplicate:
Two samples taken from and representative of the same population and carried through all steps of the sampling and analytical procedures in an identical manner. Duplicate samples are used to assess variance of the total method including sampling and analysis. (EPA-QAD)

Second Order Polynomial Curve (Quadratic): The 2nd order curves are a mathematical calculation of a slightly curved line over two axis. The y axis represents the instrument response (or Response ratio) of a standard or sample and the x axis represents the concentration. The 2nd order regression generates a coefficient of determination (COD or r^2) that is a measure of the "goodness of fit" of the quadratic curvature the data. A value of 1.00 indicates a perfect fit. In order to be used for quantitative purposes, r^2 must be greater than or equal to 0.99.

Selectivity:
(Analytical chemistry) the capability of a test method or instrument to respond to a target substance of constituent in the presence of non-target substances. (EPA-QAD)

Sensitivity:
The capability of a method or instrument to discriminate between measurement responses representing different levels (e.g., concentrations) of a variable of interest. (NELAC)

Spike:
A known mass of target analyte added to a blank, sample or sub-sample; used to determine recovery efficiency or for other quality control purposes.

If the mandated or requested test method does not specify the spiking components, the laboratory spikes all reportable components to be reported in the Laboratory Control Sample and Matrix Spike. However, in certain cases where the components interfere with accurate assessment (such as simultaneously spiking chlordane, toxaphene and PCBs in Method 608), the test method has an extremely long list of components or components are incompatible, a representative number (at a minimum 10%) of the listed components are used to control the test method. The selected components of each spiking mix must represent all chemistries, elution patterns and masses permit specified analytes and other client requested components. However, the laboratory must also ensure that all reported components are used in the spike mixture within a two-year time period. (NELAC)

Standard:
The document describing the elements of laboratory accreditation that has been developed and established within the consensus principles of NELAC and meets the approval requirements of NELAC procedures and policies. (ASQC)

Standard Operating Procedures (SOPs):
A written document which details the method of an operation, analysis, or action whose techniques and procedures are thoroughly prescribed and which is accepted as the method for performing certain routine or repetitive tasks. (QAMS)

Standardized Reference Material (SRM):
A certified reference material produced by the U.S. National Institute of Standards and Technology or other equivalent organization and characterized for absolute content, independent of analytical method. (EPA-QAD)

Supervisor (however named):
The individual(s) designated as being responsible for a particular area or category of scientific analysis. This responsibility includes direct day-to-day supervision of technical employees, supply and instrument adequacy and upkeep, quality assurance/quality control duties, and

ascertaining that technical employees have the required balance of education, training and experience to perform the required analyses. (NELAC)

Surrogate:
A substance with properties that mimic the analyte of interest, but is unlikely to be found in environment samples and is added to them for quality control purposes.

Surrogate compounds must be added to all samples, standards, and blanks, for all organic chromatography methods except when the matrix precludes its use or when a surrogate is not available. Poor surrogate recovery may indicate a problem with sample composition and is reported to the client whose sample produced poor recovery. (QAMS)

Systems Audit (also Technical Systems Audit):
A thorough, systematic, qualitative on-site assessment of the facilities, equipment, personnel, training, procedures, record keeping, data validation, data management, and reporting aspects of a total measurement system. (EPA-QAD)

Technical Director:
Individuals(s) who has overall responsibility for the technical operation of the environmental testing laboratory. (NELAC)

Test:
A technical operation that consists of the determination of one or more characteristics or performance of a given product, material, equipment, organism, physical phenomenon, process, or service according to a specified procedure. The result of a test is normally recorded in a document sometimes called a test report or a test certificate. (ISO/IEC Guide 2-12.1, amended)

Test Method:
An adoption of a scientific technique for a specific measurement problem, as documented in a laboratory SOP. (NELAC)

Toxic Substances Control Act (TSCA):
The enabling legislation in 15 USC 2601 et seq., (1976) that provides for testing, regulating, and screening all chemicals produced or imported into the United States for possible toxic effects prior to commercial manufacture. (NELAC)

Traceability:
The property of a result of a measurement whereby it can be related to appropriate standards, generally international or national standards, through an unbroken chain of comparisons. (VIM-6.12)

Uncertainty:
A parameter associated with the result of a measurement that characterizes the dispersion of the value that could reasonably be attributed to the measured value.

Acronyms:

BS – Blank Spike
BSD – Blank Spike Duplicate
CAR – Corrective Action Report
CCB – Continuing Calibration Blank

CCV – Continuing Calibration Verification
CF – Calibration Factor
CFR – Code of Federal Regulations
COC – Chain of Custody
CQMP – Corporate Quality Managemetn Plan
CRS – Change Request Form
DOC – Demonstration of Capability
DQO – Data Quality Objectives
DU – Duplicate
DUP - Duplicate
EHS – Environment, Health and Safety
EPA – Environmental Protection Agency
GC - Gas Chromatography
GC/MS - Gas Chromatography/Mass Spectrometry
HPLC - High Performance Liquid Chromatography
ICB – Initial Calibration Blank
ICP - Inductively Coupled Plasma Atomic Emission Spectroscopy
ICV – Initial Calibration Verification
IDL – Instrument Detection Limit
IH – Industrial Hygiene
IS – Internal Standard
LCS – Laboratory Control Sample
LCSD – Laboratory Control Sample Duplicate
LIMS – Laboratory Information Management System
MB – Method Blank
MDL – Method Detection Limit
MRL – Method Reporting Limit
MS – Matrix Spike
MSD – Matrix Spike Duplicate
MSDS - Material Safety Data Sheet
NELAC - National Environmental Laboratory Accreditation Conference
NELAP - National Environmental Laboratory Accreditation Program
PT – Performance Testing
QAM – Quality Assurance Manual
QA/QC – Quality Assurance / Quality Control
QAPP – Quality Assurance Project Plan
QL – Quantitation Limit
RF – Response Factor
RL – Report Limit
RPD – Relative Percent Difference
RSD – Relative Standard Deviation
SD – Standard Deviation
SOP: Standard Operating Procedure
TAT – Turn-Around-Time
VOA – Volatiles
VOC – Volatile Organic Compound

See Controlled Document QAF-45, TestAmerica Nashville Acronyms, Keywords, and Definitions.

Laboratory Certifications, Accreditations, Validations

TestAmerica Nashville maintains certifications, accreditations, certifications, and validations with numerous state and national entities. Programs vary but may include on-site audits, reciprocal agreements with another entity, performance testing evaluations, review of the QA Manual, Standard Operating Procedures, Method Detection Limits, training records, etc. At the time of this QA Manual revision, the laboratory has accreditation/certification/licensing with the following organizations:

Organization	Lab Number	Organization	Lab Number
A2LA	0453-07	Mississippi	none
AIHA	100790	Montana	none
Alabama	41150	Nevada	TN00032
Alaska	UST-087	New Hampshire (2nd NELAC)	2963
Arkansas	88-0737	New Jersey (2nd NELAC)	TN965
Arizona	AZ0473	New York (2nd NELAC)	11342
California	01168CA	North Carolina	387
Colorado	none	North Dakota	R-146
Connecticut	PH-0220	Oklahoma	9412
USACE	none	Oregon (2nd NELAC)	TN200001
Florida (1° NELAC)	E87358	Pennsylvania (2nd NELAC)	68-00585
Illinois (2nd NELAC)	200010	Rhode Island	LAO00268
Iowa	131	South Carolina	84009001, 002
Kansas (2nd NELAC)	E-10229	Tennessee	02008
Kentucky	90038	Texas (2nd NELAC)	T104704077
Kentucky UST	19	Utah (2nd NELAC)	6157260177
Louisiana Drinking H_2O	LA080021	Virginia	00323
Louisiana (2nd NELAC)	01945	Washington	C1712
Maryland	316	West Virginia	219
Massachusetts	M-TN032	Wisconsin	998020430
Minnesota	047-999-345	Wyoming UST	A2LA 0453-07

The certificates and parameter lists (which may differ) for each organization may be found on the corporate web site, the laboratory's public server, the final report review table, and in the following offices: QA, marketing, and project management.

Claims of Accreditation Status

TestAmerica Nashville has agreed to make only valid claims as to its accreditation/certification status by any authority by ensuring that the expiration dates are not exceeded and the method-specific scope or parameter lists are supportable, as required by each. Any false claims would be reported to that authority. The agreement covers the use of the authority's name, such as "Authority-Accredited," logo, or certificate number. The only valid proof of accreditation/certification is the current certificate and scope of the authority. It is the responsibility of the laboratory to make these documents available to all staff, and it is the staff's duty to reference only the current documents.

A report with scope and non-scope analytes may only be presented on the same report if the non-accredited results are clearly and unambiguously identified. No report with non-scope analytes may be associated with the logo, "Authority accredited" phrase, or the certificate number. Only the analytes specified by a unique method are valid within the scope. There must be no intentional misleading of the users of the laboratory's services in this regard.

No opinions and/or interpretations based on results outside the laboratory's scope may be presented on a document referenced by "Authority-accredited, the logo, or the certificate number. If these are made, they must be written in a separate letter which is not endorsed by the authority.

The "Authority-accredited" logo may only be affixed to equipment calibrated by a laboratory that is accredited by the authority. If calibration labels contain the logo, they must also show the calibration laboratory's name or its certificate number, the instrument's unique identification, the date of the last calibration, and a cross-reference to the last calibration certificate.

If the company decides to use the "Authority-accredited" logo in marketing activities, no misrepresentation may occur. Only reference to the accredited scope at a specific laboratory site is allowed. If any "Authority-accredited" language is used in proposals or quotations, any non-scope analytes must be clearly denoted as not accredited by that authority. The same is true for any use of laboratory letterhead with the "Authority-accredited" wording or logo. The logo may not be affixed to any material, item, product, part, or packaging, thereby implying accreditation status to that piece. In literature, any use of the logo must be positioned adjacent to the accredited laboratory's name and clearly state that the presence of the logo does not imply certification/approval of the products tested. At no time may the logo appear to suggest that a person is accredited. Misrepresentation of accreditation status is never allowed and must be reported if it occurs. If in doubt, the idea of the logo's use may be presented to the authority for approval.

If accreditation is terminated or suspended, the laboratory must immediately cease to use the "Authority-accredited" wording, the logo, or the certificate number reference in any way and inform clients impacted by the change.

Data Qualifiers

<	< [Custom Value]
>	> [Custom Value]
A-01	[Custom Value]
B	Analyte was detected in the associated Method Blank.
B1	Analyte was detected in the associated method blank. Analyte concentration in the sample is greater than 10x the concentration found in the method blank.
B7	Analyte detected in refrigerator/freezer holding blank. Reported value for this sample within 10x detected concentration in holding blank.
BCD	The contamination reported in the HPLC method blank for one detector was not confirmed by the other detector.
C	Calibration Verification recovery was above the method control limit for this analyte. Analyte not detected, data not impacted.
C1	Calibration Verification recovery was above the method control limit for this analyte, however the average % difference for all analytes met method criteria.
C2	Calibration Verification recovery was below the method control limit for this analyte, however the average % difference for all analytes met method criteria.
C4	Calibration Verification recovery was below the method control limit for this analyte.
C8	Calibration Verification recovery was above the method control limit for this analyte. A high bias may be indicated.
CF2	Confirmatory analysis was past holding time.
CF5	The sample was originally analyzed with a positive result, however the reanalysis did not confirm the presence of the analyte.
CF6	Results confirmed by reanalysis.
CF7	Result may be elevated due to carry over from previously analyzed sample.
CISP	The concentration indicated for this analyte is derived from a single point calibration.
CL1	Recovery for this analye was within Laboratory historical limits but outside contract required limits of 70-130 %.
CN4	Amenable cyanide results reported from total determination method.
CSTM	[Custom Value]
E	Concentration exceeds the calibration range and therefore result is semi-quantitative.
E1	Concentration estimated. Analyte exceeded calibration range. Reanalysis not possible due to insufficient sample remaining.
E3	Concentration estimated. Analyte exceeded calibration range. Reanalysis not peformed due to holding time requirements.
EPH%	[Custom Value] %RPD
H	Sample analysis performed past method-specified holding time.
H1	Sample analysis performed past the method-specified holding time per client's approval.
H10	The holding time calculation is based on a sampling time of 00:00 on the sampling date noted on the Chain of Custody. No sampling time was provided to the laboratory.
H2	Initial analysis within holding time. Reanalysis for the required dilution or confirmation was past holding time.
H3	Sample was received and analyzed past holding time.

H4	Sample was extracted past holding time, but analyzed within analysis holding time.
H8	The sample was extracted past the holding time.
HT2	Received out of 48 hour hold for 5035 preparation.
HT3	Sample received with insufficient holding time remaining for analysis to be performed within the method's holding time requirements.
HTI	The holding time for this test is immediate. The laboratory measurement, therefore, may not be suitable for compliance purposes.
I	Internal Standard recovery was outside of method limits. Matrix interference was confirmed by reanalysis.
ID2	Secondary ion abundances were outside method requirements. Identification based on analytical judgement.
IH1	Target analyte was detected in the method blank at or above the reporting limit. The background was subtracted from the QC samples and the client's sample(s).
IH10	Target analyte was not detected in the method blank at or above the reporting limit. The QC samples and client sample(s) were not blank corrected.
IH2	Target analyte was detected in the method blank at or above the reporting limit. The background was not subtracted from the client's sample(s).
IH3	Target analyte was detected in the client's field blank at or above the reporting limit. The background was subtracted from the client's sample(s).
IH4	Target analyte was detected in the client's field blank at or above the reporting limit. The background was not subtracted from the client's sample(s).
IH8	Target analyte was detected in the method blank below the reporting limit but above the MDL. The background was subtracted from the QC samples and the client's sample(s).
J	Analyte detected at a level less than the Reporting Limit (RL) and greater than or equal to the Method Detection Limit (MDL). Concentrations within this range are estimated.
K	The sample dilutions set-up for the BOD analysis did not meet the oxygen depletion criteria of at least 2 mg/l. Therefore the reported result is an estimated value only.
K1	The sample dilutions set up for the BOD analysis failed to meet the criteria of a residual dissolved oxygen of at least 1 mg/L. Therefore the reported result is an estimated value only.
K2	The seed depletion was outside the method acceptance limits. Therefore, the reported result is an estimated value only.
K3	The dilution water D.O. depletion was > 0.2 mg/L.
K4	The seed depletion was not within method recommended limits. The LCS, which is a means of checking dilution water quality and seed effectiveness, was within acceptance limits.
K5	Residual chlorine detected. Sample dechlorinated prior to analysis.
L	Laboratory Control Sample and/or Laboratory Control Sample Duplicate recovery was above the acceptance limits. Analyte not detected, data not impacted.
L1	Laboratory Control Sample and/or Laboratory Control Sample Duplicate recovery was above acceptance limits.
L2	Laboratory Control Sample and/or Laboratory Control Sample Duplicate recovery was below acceptance limits.
L4	Laboratory Control Sample and/or Laboratory Control Sample Duplicate recovery was below the acceptance limits. A low bias to sample results is indicated.
M1	The MS and/or MSD were above the acceptance limits due to sample matrix interference. See Blank Spike (LCS).

M10	Matrix Spike recovery was low. Data Reported per ADEQ policy 0154.000
M2	The MS and/or MSD were below the acceptance limits due to sample matrix interference. See Blank Spike (LCS).
M3	Results exceeded the linear range in the MS/MSD and therefore are not available for reporting. The batch was accepted based on acceptable recovery in the Blank Spike (LCS).
M4	The MS/MSD required a dilution due to matrix interference. Because of this dilution, the matrix spike concentrations in the sample were reduced to a level where the recovery calculation does not provide useful information. See Blank Spike (LCS).
M6	Any analyte not run due to matrix
M7	The MS and/or MSD were above the acceptance limits. See Blank Spike (LCS).
M8	The MS and/or MSD were below the acceptance limits. See Blank Spike (LCS).
M9	Matrix Spike recovery was high. Data Reported per ADEQ policy 0154.000
MCP	No results were reported for the MS and/or MSD due to a clogged autosampler port. Batch was accepted based on Blank Spike (LCS) recoveries.
MHA	Due to high levels of analyte in the sample, the MS/MSD calculation does not provide useful spike recovery information. See Blank Spike (LCS).
MNR	No results were reported for the MS/MSD. The sample used for the MS/MSD required dilution due to the sample matrix. Because of this, the spike compounds were diluted below the detection limit.
MNR1	There was no MS/MSD analyzed with this batch due to insufficient sample volume. See Blank Spike.
MNR3	Insufficient sample received to meet method QC requirements.
MNR4	Insufficient sample received to meet method QC requirements. Batch QC requirements satisfy ADEQ policies 0154.000 and 0155.000
N1	See case narrative.
N2	See corrective action report.
NO	No
NoRES	>100,000
P	The sample, as received, was not preserved in accordance to the referenced analytical method.
P1	Sample received and analyzed without chemical preservation.
P12	The method required trip blank was not provided along with this sample.
P2	Sample received without chemical preservation, but preserved by the laboratory.
P3	Sample was received above recommended temperature.
P4	Sample received in inappropriate sample container.
P6	Sample received unpreserved, however the sample was analyzed within 7 days per EPA recommendation.
P7	Sample filtered in lab.
P8	Sample unable to be adjusted to correct pH due to matrix.
P9	This analyte has been shown to degrade upon preservation with HCl and cannot accurately be quantitated.
pH	pH [Custom Value]
pH<1	pH<1
pH<2	pH<2
pH>12	pH>12
pH>13	pH>13

pH>2	pH>2
P-HS	Sample container contained headspace.
PV	Acid preservation was indicated on the sample vial. However, a pH of <2 was not obtained.
PX	Sample for VOA analysis not received in preserved VOA vials or Encore or similar sampling device.
Q2	The chromatographic pattern is consistent with diesel fuel.
Q3	The chromatographic pattern is not consistent with diesel fuel.
Q5	Results in the diesel organics range are primarily due to overlap from a gasoline range product.
QFL	Florisil clean-up (EPA 3620) performed on extract.
QGP	Gel Permeation (EPA 3640) clean-up performed on extract.
QP	Hydrocarbon result partly due to individual peak(s) in quantitation range.
QP1	The primary contamination elutes between [Custom Value], which is in the motor oil range.
QP2	The primary contamination elutes between [Custom Value], which is in the diesel fuel range.
QP3	The primary contamination elutes between [Custom Value], which is in the kerosene range.
QP4	The primary contamination elutes between [Custom Value], which is in the mineral spirits range.
QP5	There was insufficient contamination present to perform a pattern match.
QP6	The contamination did not match any standards in our library.
QP7	The contamination is similar to our [Custom Value] standard.
QSG	Silica Gel clean-up performed on extracts.
QSP	Sulfuric Acid / Permanganate (EPA3665) clean-up performed on extract.
QSU	Sulfur (EPA 3660) clean-up performed on extract.
QU	Unquantitated hydrocarbons present in the sample outside of the reported carbon range.
R	The RPD exceeded the method control limit. The individual analyte QA/QC recoveries, however, were within acceptance limits.
R1	The RPD between the primary and confirmatory analysis exceeded 40%. Per method 8000B, the higher value was reported.
R10	The RPD between the primary and confirmatory analysis exceeded 40%. Per method 8000B, the lower value was reported due to apparent chromatographic problems.
R12	The RPD between the primary and confirmatory analysis exceeded 40%. Per method 8000C, the lower value was reported.
R2	The RPD exceeded the acceptance limit.
R3	The RPD exceeded the acceptance limit due to sample matrix effects.
R4	Due to the low levels of analyte in the sample, the duplicate RPD calculation does not provide useful information.

R7	LCS/LCSD RPD exceeded the acceptance limit. Recovery met acceptance criteria.
R9	Sample RPD exceeded the laboratory control limit.
RL1	Reporting limit raised due to sample matrix effects.
RL4	Reporting limit raised due to insufficient sample volume.
S10	Insufficient sample available for reanalysis.
S12	Sample highly flammable and ignited upon contact.
S6	Sediment present.
S7	Sample breakthrough to 2nd section is > 10%. Results may be biased low.
SB	Sustained burning when exposed to open flame.
SPS	Percent solids result provided by the client.
SSV	Solvent volume not present on sample vial. The lab has assumed a 1:1 ratio.
STW	No tare weight present on sample vial. Result should be considered an estimated value.
T1	Method approved by EPA, but not yet licensed by ADHS.
T2	Cited ADHS licensed method does not contain this analyte as part of method compound list.
T4	The cited licensed method does not contain this analyte as part of the method compound list.
T5	Less than the prescribed sample amount was available to perform the leachate extraction. The volume of extraction fluid was adjusted proportionately based on the method prescribed ratio of extraction fluid to sample weight.
T6	The temperature during the 18 hour TCLP extraction exceeded the 21-25 degrees C range stated in EPA Method 1311. The temperature range during the extraction was [Custom Value] degrees C.
T7	Tentatively identified compound. Concentration is estimated based on the closest internal standard.
T8	The reported result cannot be used for compliance purposes.
TND	Compound not detected using TIC procedure. Quantitation is estimated based on closest Internal Standard. An assumption is made that the compound will purge or extract and respond chromatographically consistent with target compounds analyzed by this method.
YES	Yes
Z	Due to sample matrix effects, the surrogate recovery was below the acceptance limits.
Z1	Surrogate recovery was above acceptance limits.
Z10	Surrogate outside laboratory historical limits but within method guidelines. No effect on data.
Z11	Surrogate low but all targets within method criteria. No effect on data.
Z2	Surrogate recovery was above the acceptance limits. Data not impacted.
Z3	The sample required a dilution due to the nature of the sample matrix. Because of this dilution, the surrogate spike concentration in the sample was reduced to a level where the recovery calculation does not provide useful information.
Z5	Due to sample matrix effects, the surrogate recovery was outside acceptance limits. Secondary surrogate recovery was within the acceptance limits.
Z6	Surrogate recovery was below acceptance limits.

Z7	Surrogate recovery was high. Data reported per ADEQ policy 0154.000.
Z8	Surrogate recovery was low. Data reported per ADEQ policy 0154.000.
ZE	The EPH surrogate recovery was outside QC limits due to poor fractionation. Both fractions were evaluated for all EPH carbon ranges. The sample was non-detect at the reporting limit for all carbon ranges, so the data was accepted without further analysis.
ZX	Due to sample matrix effects, the surrogate recovery was outside the acceptance limits.

For Ohio VAP: The laboratory must implement corrective action procedures to resolve the deviation and limit qualification of the final results. The laboratory is not permitted to deviate from its VAP approved SOP if it intends to attest under affidavit that the "results" are VAP certified. When all corrective actions listed in the SOP have been exhausted, it may be necessary to use technical judgment in which case the decision process and rationale is presented in the final report and/or affidavit and the data will be noted as 'not VAP certified' on the affidavit.

Federal Appendix I - Constituents for Assessment Monitoring (40 CFR Part 258)

Appendix I Constituents	CAS Registry Number	Maximum Contaminant Level (mg/L)	RCRA Method
VOLATILES			
Acetone	67-64-1		8260B
Acrylonitrile	107-13-1		8260B
Benzene	71-43-2	0.005	8260B
Bromochloromethane; Chlorobromomethane	74-97-5		8260B
Bromodichloromethane; Dichlorobromomethane	75-27-4	**	8260B
Bromoform; Tribromomethane	75-25-5	**	8260B
Carbon disulfide	75-15-0		8260B
Carbon tetrachloride	56-23-5	0.005	8260B
Chlorobenzene	108-90-7	0.1	8260B
Chloroethane; Ethyl chloride	75-00-3		8260B
Chloroform; Trichloro-methane	67-66-3	**	8260B
Dibromochloromethane; Chlorodibromomethane	124-48-1	**	8260B
1,2-Dibromo-3-chloropro-pane; DBCP	96-12-8	0.0002	8011
1,2-Dibromoethane; Ethylene dibromide; EDB	106-93-4	0.00005	8011
o-Dichlorobenzene; 1,2-Dichlorobenzene	95-50-1	0.6	8260B
p-Dichlorobenzene; 1,4-Dichlorobenzene	106-46-7	0.075	8260B
trans-1,4-Dichloro-2-butene	110-57-6		8260B
1,1-Dichloroethane; Ethylidene chloride	75-34-3		8260B
1,2-Dichloroethane; Ethylene dichloride	107-06-2	0.005	8260B
1,1-Dichloroethylene; Vinylidene chloride; 1,1-Dichloroethene	75-35-4	0.007	8260B
cis-1,2-Dichloroethylene; cis-1,2-Dichloroethene	156-59-2	0.07	8260B
trans-1,2-Dichloroethylene; trans-1,2-Dichloroethene	156-60-5	0.1	8260B
1,2-Dichloropropane; Propylene dichloride	78-87-5	0.005	8260B
cis-1,3-Dichloropropene	10061-01-5		8260B
trans-1,3-Dichloropropene	10061-02-6		8260B
Ethylbenzene	100-41-4	0.7	8260B
2-Hexanone; Methyl butyl ketone	591-78-6		8260B
Methyl bromide; Bromomethane	74-83-9		8260B
Methyl chloride; Chloromethane	74-87-3		8260B
Methyl ethyl ketone; MEK; 2-Butanone	78-93-3		8260B
Methyl iodide; Iodomethane	74-88-4		8260B
4-Methyl-2-pentanone; Methyl isobutyl ketone	108-10-1		8260B
Methylene chloride; Dichloromethane	75-09-2	0.005	8260B
Styrene	100-42-5	0.1	8260B
1,1,1,2-Tetrachloroethane	630-20-6		8260B
1,1,2,2-Tetrachloroethane	79-34-5		8260B
Tetrachloroethylene; Tetrachloroethene; Perchloroethylene	127-18-4	0.005	8260B
Toluene	108-88-3	1	8260B
1,1,1-Trichloroethane; Methyl chloroform	71-55-6	0.2	8260B
1,1,2-Trichloroethane	79-00-5	0.005	8260B
Trichloroethylene; Trichloroethene	79-01-6	0.005	8260B

Appendix I Constituents	CAS Registry Number	Maximum Contaminant Level (mg/L)	RCRA Method
Trichlorofluoromethane	75-69-4		8260B
1,2,3-Trichloropropane	96-18-4		8260B
Vinyl acetate	108-05-4		8260B
Vinyl chloride; Chloroethene	75-01-4	0.002	8260B
Xylene (total)	96-47-6, 108-38-3, 106-42-3, 1330-20-7.	10	8260B
METALS			
Antimony	7440-36-0	0.006	6010B
Arsenic	7440-38-2	0.05	6010B
Barium	7440-39-3	2.0	6010B
Beryllium	7440-41-7	0.004	6010B
Cadmium	7440-43-9	0.005	6010B
Chromium	7440-47-3	0.1	6010B
Cobalt	7440-48-4		6010B
Copper	7440-50-8	1	6010B
Lead	7439-92-1	0.015	6010B
Nickel	7440-02-0	0.1	6010B
Selenium	7782-49-2	0.05	6010B
Silver	7440-22-4	0.1	6010B
Thallium	7440-28-0	0.002	6010B
Vanadium	7440-62-2		6010B
Zinc	7440-66-6	5	6010B

Note: Depending upon the state, additional constituents may be required.

Federal Appendix II - Constituents for Assessment Monitoring (40 CFR Part 258)

Appendix II Constituents	CAS Registry Number	Maximum Contaminant Level (mg/L)	RCRA Method
VOLATILES			
Acetone	67-64-1		8260B
Acetonitrile; Methyl cyanide	75-05-8		8260B
Acrolein	107-02-8		8260B
Acrylonitrile	107-13-1		8260B
Allyl chloride	107-05-1		8260B
Benzene	71-43-2	0.005	8260B
Bromochloromethane; Chlorobromomethane	74-97-5		8260B
Bromodichloromethane; Dichlorobromomethane	75-27-4	**	8260B
Bromoform; Tribromomethane	75-25-5	**	8260B
Carbon disulfide	75-15-0		8260B
Carbon tetrachloride	56-23-5	0.005	8260B
Chlorobenzene	108-90-7	0.1	8260B
Chloroethane; Ethyl chloride	75-00-3		8260B
Chloroform; Trichlorome-thane	67-66-3	**	8260B
Chloroprene	126-99-8		8260B
Dibromochloromethane; Chlorodibromomethane	124-48-1	**	8260B
1,2-Dibromo-3-chloropro-pane; DBCP	96-12-8	0.0002	8011
1,2-Dibromoethane; Ethylene dibromide; EDB	106-93-4	0.00005	8011
o-Dichlorobenzene; 1,2-Dichlorobenzene	95-50-1	0.6	8260B
m-Dichlorobenzene; 1,3-Dichlorobenzene	541-73-1		8260B
p-Dichlorobenzene; 1,4-Dichlorobenzene	106-46-7	0.075	8260B
trans-1,4-Dichloro-2-butene	110-57-6		8260B
Dichlorodifluoromethane	75-71-8		8260B
1,1-Dichloroethane; Ethylidene chloride	75-34-3		8260B
1,2-Dichloroethane; Ethylene dichloride	107-06-2	0.005	8260B
1,1-Dichloroethylene; Vinylidene chloride; 1,1-Dichloroethene	75-35-4	0.007	8260B
cis-1,2-Dichloroethylene; cis-1,2-Dichloroethene	156-59-2	0.07	8260B
trans-1,2-Dichloroethylene; trans-1,2-Dichloroethene	156-60-5	0.1	8260B
1,2-Dichloropropane; Propylene dichloride	78-87-5	0.005	8260B
1,3-Dichloropropane; Trimethylene dichloride	142-28-9		8260B
2,2-Dichloropropane; Isopropylidene chloride	594-20-7		8260B
1,1-Dichloropropene	563-58-6		8260B
cis-1,3-Dichloropropene	10061-01-5		8260B
trans-1,3-Dichloropropene	10061-02-6		8260B
Ethylbenzene	100-41-4	0.7	8260B
Ethyl methacrylate	97-63-2		8260B
2-Hexanone; Methyl butyl ketone	591-78-6		8260B
Isobutyl alcohol	78-83-1		8260B
Methacrylonitrile	126-98-7		8260B
Methyl bromide; Bromomethane	74-83-9		8260B
Methyl chloride; Chloromethane	74-87-3		8260B
Methyl ethyl ketone; MEK; 2-Butanone	78-93-3		8260B
Methyl iodide; Iodomethane	74-88-4		8260B
Methyl methacrylate	80-62-6		8260B

Appendix II Constituents	CAS Registry Number	Maximum Contaminant Level (mg/L)	RCRA Method
4-Methyl-2-pentanone; Methyl isobutyl ketone	108-10-1		8260B
Methylene bromide; Dibromomethane	74-95-3		8260B
Methylene chloride; Dichloromethane	75-09-2	0.005	8260B
Propionitrile; Ethyl cyanide	107-12-0		8260B
Styrene	100-42-5	0.1	8260B
1,1,1,2-Tetrachloroethane	630-20-6		8260B
1,1,2,2-Tetrachloroethane	79-34-5		8260B
Tetrachloroethylene; Tetrachloroethene; Perchloroethylene	127-18-4	0.005	8260B
Toluene	108-88-3	1	8260B
1,1,1-Trichloroethane; Methyl chloroform	71-55-6	0.2	8260B
1,1,2-Trichloroethane	79-00-5	0.005	8260B
Trichloroethylene; Trichloroethene	79-01-6	0.005	8260B
Trichlorofluoromethane	75-69-4		8260B
1,2,3-Trichloropropane	96-18-4		8260B
Vinyl acetate	108-05-4		8260B
Vinyl chloride; Chloroethene	75-01-4	0.002	8260B
Xylene (total)	96-47-6, 108-38-3, 106-42-3, 1330-20-7.	10	8260B
SEMIVOLATILES			
Acenaphthene	83-32-9		8270C
Acenaphthylene	208-96-8		8270C
Acetophenone	98-86-2		8270C
2-Acetylaminofluorene; 2-AAF	53-96-3		8270C
4-Aminobiphenyl	92-67-1		8270C
Anthracene	120-12-7		8270C
Benzo[a]anthracene; Benzathracene	56-55-3		8270C
Benzo[b]fluoranthene	205-99-2		8270C
Benzo[k]fluoranthene	207-08-9		8270C
Benzo[ghi]perylene	191-24-2		8270C
Benzyl alcohol	100-51-6		8270C
Bis(2-chloroethoxy)methane	111-91-1		8270C
Bis(2-chloroethyl)ether; Dichloroethyl ether	111-44-4		8270C
Bis(2-chloroisopropyl)ether; Bis(2-chloro-1-methylethyl) ether; 2,2-Dichlorodiisopro-pyl ether; DCIP	108-60-1		8270C
Bis(2-ethylhexyl) phthalate	117-81-7	0.006	8270C
4-Bromophenyl phenyl ether	101-55-3		8270C
Butyl benzyl phthalate	85-68-7		8270C
p-Chloroaniline; 4-Chloro-aniline	106-47-8		8270C
Chlorobenzilate	510-15-6		8270C
p-Chloro-m-cresol; 4-Chloro-3-methylphenol	59-50-7		8270C
2-Chloronaphthalene	91-58-7		8270C
2-Chlorophenol	95-57-8		8270C
4-Chlorophenyl phenyl ether	7005-72-3		8270C
Chrysene	218-01-9		8270C
m-Cresol; 3-Methylphenol	108-39-4		8270C
o-Cresol; 2-Methylphenol	95-48-7		8270C

Appendix II Constituents	CAS Registry Number	Maximum Contaminant Level (mg/L)	RCRA Method
p-Cresol; 4-Methylphenol	106-44-5		8270C
Diallate	2303-16-4		8270C
Dibenz[a,h]anthracene	53-70-3		8270C
Dibenzofuran	132-64-9		8270C
Di-n-butyl phthalate	84-74-2		8270C
3,3'-Dichlorobenzidine	91-94-1		8270C
2,4-Dichlorophenol	120-83-2		8270C
2,6-Dichlorophenol	87-65-0		8270C
Diethyl phthalate	84-66-2		8270C
o,o-Diethyl-o-2-pyrazinyl phosphorothioate; Thionazin	297-97-2		8270C
Dimethoate	60-51-5		8270C
p-(Dimethylamino)azoben-zene	60-11-7		8270C
7,12-Dimethylbenz[a]an-thracene	57-97-6		8270C
3,3'-Dimethylbenzidine	119-93-7		8270C
2,4-Dimethylphenol	105-67-9		8270C
Dimethyl phthalate	131-11-3		8270C
m-Dinitrobenzene	99-65-0		8270C
4,6-Dinitro-o-cresol; 4,6-Dinitro-2-methylphenol	534-52-1		8270C
2,4-Dinitrophenol	51-28-5		8270C
2,4-Dinitrotoluene	121-4-2		8270C
2,6-Dinitrotoluene	606-20-2		8270C
Di-n-octyl phthalate	117-84-0		8270C
Diphenylamine	122-39-4		8270C
Disulfoton	298-04-4		8270C
Ethyl methanesulfonate	62-50-0		8270C
Famphur	52-85-7		8270C
Fluoranthene	206-44-0		8270C
Fluorene	86-73-7		8270C
Hexachlorobenzene	118-74-1	0.001	8270C
Hexachlorobutadiene	87-68-3		8270C
Hexachlorocyclopentadiene	77-47-4	0.05	8270C
Hexachloroethane	67-72-1		8270C
Hexacloroproprene	1888-71-7		8270C
Indeno[1,2,3-cd]pyrene	193-39-5		8270C
Isodrin	465-73-6		8270C
Isophorone	78-59-1		8270C
Isosafrole	120-58-1		8270C
Kepone	143-50-0		8270C
Methapyrilene	91-80-5		8270C
3-Methylcholanthrene	56-49-5		8270C
Methyl methanesulfonate	66-27-3		8270C
2-Methylnaphthalene	91-57-6		8270C
Methyl parathion; Parathion methyl	298-00-0		8270C
Naphthalene	91-20-3		8270C
1,4-Naphthoquinone	130-15-4		8270C
1-Naphthylamine	134-32-7		8270C
2-Naphthylamine	91-59-8		8270C
o-Nitroaniline; 2-Nitroaniline	88-74-4		8270C

Appendix II Constituents	CAS Registry Number	Maximum Contaminant Level (mg/L)	RCRA Method
m-Nitroaniline;3-Nitroaniline	99-09-2		8270C
p-Nitroaniline; 4-Nitroaniline	100-01-6		8270C
Nitrobenzene	98-95-3		8270C
o-Nitrophenol; 2-Nitrophenol	88-75-5		8270C
p-Nitrophenol; 4-Nitrophenol	100-02-7		8270C
N-nitrosodi-n-butylamine	924-16-3		8270C
N-nitrosodiethylamine	55-18-5		8270C
N-nitrosodimethylamine	62-75-9		8270C
N-nitrosodiphenylamine	86-30-6		8270C
N-nitrosodipropylamine; Di-n-propyl-nitrosamine; N-nitroso-n-dipropylamine	621-64-7		8270C
N-nitrosomethylethylamine	1059-95-6		8270C
N-nitrosopiperidine	100-75-4		8270C
N-nitrosopyrrolidine	930-55-2		8270C
5-Nitro-o-toluidine	99-55-8		8270C
Parathion	56-38-2		8270C
Pentachlorobenzene	608-93-5		8270C
Pentachloronitrobenzene	82-68-8		8270C
Phenacetin	62-44-2		8270C
Phenanthrene	85-01-8		8270C
Phenol	108-95-2		8270C
p-Phenylenediamine	106-50-3		8270C
Phorate	298-02-2		8270C
Pronamide	23950-58-5		8270C
Pyrene	129-00-0		8270C
Safrole	94-59-7		8270C
1,2,4,5-Tetrachlorobenzene	95-94-3		8270C
2,3,4,6-Tetrachlorophenol	58-90-2		8270C
o-Toluidine	95-53-4		8270C
1,2,4-Trichlorobenzene	120-82-1		8270C
2,4,5-Trichlorophenol	95-95-4		8270C
2,4,6-Trichlorophenol	88-06-2		8270C
o,o,o-Triethyl phosphoro-thioate; Terbufos	126-68-1		8270C
sym-Trinitrobenzene; 1,3,5-Trinitrobenzene	99-35-4		8270C
Benzo[a]pyrene	50-32-8	0.0002	8310
ORGANOCHLORINE PESTICIDES/PCBs			
Aldrin	309-00-2		8081A
alpha-BHC; alpha-Benzene hexachloride	319-84-6		8081A
beta-BHC; beta-Benzene hexachloride	309-85-7		8081A
delta-BHC;delta-Benzene hexachloride	319-86-8		8081A
gamma-BHC; gamma-Benzene hexachloride; Lindane	58-89-9	0.0002	8081A
alpha-Chlordane	5103-71-9	0.002	8081A
gamma-Chlordane	5103-74-2	0.002	8081A
4,4'-DDD	72-54-8		8081A
4,4'-DDE	72-55-9		8081A
4,4'-DDT	50-29-3		8081A
Dieldrin	60-57-1		8081A
Endosulfan I	959-98-8		8081A

Appendix II Constituents	CAS Registry Number	Maximum Contaminant Level (mg/L)	RCRA Method
Endosulfan II	33213-65-9		8081A
Endosulfan sulfate	1031-07-8		8081A
Endrin	72-20-8	0.002	8081A
Endrin aldehyde	7421-93-4		8081A
Heptachlor	76-44-8	0.0004	8081A
Heptachlor epoxide	1024-57-3	0.0002	8081A
Methoxychlor	72-43-5	0.04	8081A
Toxaphene	8001-35-2	0.003	8081A
Polychlorinated biphenyls; PCBs; Aroclors	1336-36-3, 12674-11-2, 11104-28-2, 11141-16-5, 53469-21-9, 12672-29-6, 11097-69-1, 11096-82-5	0.0005	8082
HERBICIDES			
2,4-D; 2,4-Dichlorophenoxy-acetic acid	94-75-7	0.02	8151A
2,4,5-T; 2,4,5-Trichlorophen-oxyacetic acid	93-76-5		8151A
2,4,5-TP; 2,4,5-Trichloro-phenoxypropanoic acid; Silvex	93-72-1	0.05	8151A
Dinoseb; DNBP; 2-sec-Butyl-4,6-dinitrophenol	88-85-7		8151A
Pentachlorophenol	87-86-5	0.001	8151A
METALS			
Antimony	7440-36-0	0.006	6010B
Arsenic	7440-38-2	0.05	6010B
Barium	7440-39-3	2.0	6010B
Beryllium	7440-41-7	0.004	6010B
Cadmium	7440-43-9	0.005	6010B
Chromium	7440-47-3	0.1	6010B
Cobalt	7440-48-4		6010B
Copper	7440-50-8	1	6010B
Lead	7439-92-1	0.015	6010B
Nickel	7440-02-0	0.1	6010B
Selenium	7782-49-2	0.05	6010B
Silver	7440-22-4	0.1	6010B
Thallium	7440-28-0	0.002	6010B
Tin	7440-31-5		6010B
Vanadium	7440-62-2		6010B
Zinc	7440-66-6	5	6010B
Mercury	7439-97-6	0.002	7470A
OTHER INORGANICS			
Cyanide	57-12-5	0.2 as Free	9012B
Sulfide	18496-25-8		9034

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Appendix IX Constituents	CAS Registry Number	RCRA Method
VOLATILES		
Acetone	67-64-1	8260B
Acetonitrile; Methyl cyanide	75-05-8	8260B
Acrolein	107-02-8	8260B
Acrylonitrile	107-13-1	8260B
Allyl chloride	107-05-1	8260B
Benzene	71-43-2	8260B
Bromodichloromethane; Dichlorobromomethane	75-27-4	8260B
Bromoform; Tribromomethane	75-25-5	8260B
Carbon disulfide	75-15-0	8260B
Carbon tetrachloride	56-23-5	8260B
Chlorobenzene	108-90-7	8260B
Chloroethane; Ethyl chloride	75-00-3	8260B
Chloroform; Trichloro-methane	67-66-3	8260B
Chloroprene	126-99-8	8260B
Dibromochloromethane; Chlorodibromomethane	124-48-1	8260B
1,2-Dibromo-3-chloropro-pane; DBCP	96-12-8	8011
1,2-Dibromoethane; Ethylene dibromide; EDB	106-93-4	8011
o-Dichlorobenzene; 1,2-Dichlorobenzene	95-50-1	8260B
m-Dichlorobenzene; 1,3-Dichlorobenzene	541-73-1	8260B
p-Dichlorobenzene; 1,4-Dichlorobenzene	106-46-7	8260B
trans-1,4-Dichloro-2-butene	110-57-6	8260B
Dichlorodifluoromethane	75-71-8	8260B
1,1-Dichloroethane; Ethylidene chloride	75-34-3	8260B
1,2-Dichloroethane; Ethylene dichloride	107-06-2	8260B
1,1-Dichloroethylene; Vinylidene chloride; 1,1-Dichloroethene	75-35-4	8260B
cis-1,2-Dichloroethylene; cis-1,2-Dichloroethene	156-59-2	8260B
trans-1,2-Dichloroethylene; trans-1,2-Dichloroethene	156-60-5	8260B
1,2-Dichloropropane; Propylene dichloride	78-87-5	8260B
cis-1,3-Dichloropropene	10061-01-5	8260B
trans-1,3-Dichloropropene	10061-02-6	8260B
1,4-Dioxane	123-91-1	8260B
Ethylbenzene	100-41-4	8260B
Ethyl methacrylate	97-63-2	8260B
Hexachlorobutadiene	87-68-3	8260B
2-Hexanone; Methyl butyl ketone	591-78-6	8260B
Isobutyl alcohol	78-83-1	8260B
Methacrylonitrile	126-98-7	8260B
Methyl bromide; Bromomethane	74-83-9	8260B
Methyl chloride; Chloromethane	74-87-3	8260B
Methyl ethyl ketone; MEK; 2-Butanone	78-93-3	8260B
Methyl iodide; Iodomethane	74-88-4	8260B
Methyl methacrylate	80-62-6	8260B
4-Methyl-2-pentanone; Methyl isobutyl ketone	108-10-1	8260B
Methylene bromide; Dibromomethane	74-95-3	8260B

Appendix IX Constituents	CAS Registry Number	RCRA Method
Methylene chloride; Dichloromethane	75-09-2	8260B
Propionitrile; Ethyl cyanide	107-12-0	8260B
Styrene	100-42-5	8260B
1,1,1,2-Tetrachloroethane	630-20-6	8260B
1,1,2,2-Tetrachloroethane	79-34-5	8260B
Tetrachloroethylene; Tetrachloroethene; Perchloroethylene	127-18-4	8260B
Toluene	108-88-3	8260B
1,1,1-Trichloroethane; Methyl chloroform	71-55-6	8260B
1,1,2-Trichloroethane	79-00-5	8260B
Trichloroethylene; Trichloroethene	79-01-6	8260B
Trichlorofluoromethane	75-69-4	8260B
1,2,3-Trichloropropane	96-18-4	8260B
Vinyl acetate	108-05-4	8260B
Vinyl chloride; Chloroethene	75-01-4	8260B
Xylenes (total)	96-47-6, 108-38-3, 106-42-3, 1330-20-7.	8260B
SEMIVOLATILES		
Acenaphthene	83-32-9	8270C
Acenaphthylene	208-96-8	8270C
Acetophenone	98-86-2	8270C
2-Acetylaminofluorene; 2-AAF	53-96-3	8270C
4-Aminobiphenyl	92-67-1	8270C
Aniline	62-53-3	8270C
Anthracene	120-12-7	8270C
Aramite	140-57-8	8270C
Benzo(a)anthracene; Benzathracene	56-55-3	8270C
Benzo(a)pyrene	50-32-8	8270C
Benzo(b)fluoranthene	205-99-2	8270C
Benzo(k)fluoranthene	207-08-9	8270C
Benzo(ghi)perylene	191-24-2	8270C
Benzyl alcohol	100-51-6	8270C
Bis(2-chloroethoxy)methane	111-91-1	8270C
Bis(2-chloroethyl)ether; Dichloroethyl ether	111-44-4	8270C
Bis(2-chloro-1-methylethyl) ether; 2,2-Dichlorodiiso-propyl ether; DCIP	108-60-1	8270C
Bis(2-ethylhexyl) phthalate	117-81-7	8270C
4-Bromophenyl phenyl ether	101-55-3	8270C
Butyl benzyl phthalate	85-68-7	8270C
p-Chloroaniline; 4-Chloro-aniline	106-47-8	8270C
Chlorobenzilate	510-15-6	8270C
p-Chloro-m-cresol; 4-Chloro-3-methylphenol	59-50-7	8270C
2-Chloronaphthalene	91-58-7	8270C
2-Chlorophenol	95-57-8	8270C
4-Chlorophenyl phenyl ether	7005-72-3	8270C
Chrysene	218-01-9	8270C
m-Cresol; 3-Methylphenol	108-39-4	8270C

Appendix IX Constituents	CAS Registry Number	RCRA Method
o-Cresol; 2-Methylphenol	95-48-7	8270C
p-Cresol; 4-Methylphenol	106-44-5	8270C
Diallate	2303-16-4	8270C
Dibenz(a,h)anthracene	53-70-3	8270C
Dibenzofuran	132-64-9	8270C
Di-n-butyl phthalate	84-74-2	8270C
3,3'-Dichlorobenzidine	91-94-1	8270C
2,4-Dichlorophenol	120-83-2	8270C
2,6-Dichlorophenol	87-65-0	8270C
Diethyl phthalate	84-66-2	8270C
o,o-Diethyl-o-2-pyrazinyl phosphorothioate; Thionazine	297-97-2	8270C
Dimethoate	60-51-5	8270C
p-(Dimethylamino)azobenzene	60-11-7	8270C
7,12-Dimethylbenz(a)anthracene	57-97-6	8270C
3,3'-Dimethylbenzidine	119-93-7	8270C
2,4-Dimethylphenol	105-67-9	8270C
Dimethyl phthalate	131-11-3	8270C
a,a-Dimethylphenethylamine	122-09-8	8270C
m-Dinitrobenzene; 1,3-Dinitrobenzene	99-65-0	8270C
4,6-Dinitro-o-cresol; 4,6-Dinitro-2-methylphenol	534-52-1	8270C
2,4-Dinitrophenol	51-28-5	8270C
2,4-Dinitrotoluene	121-4-2	8270C
2,6-Dinitrotoluene	606-20-2	8270C
Di-n-octyl phthalate	117-84-0	8270C
Dinoseb; DNBP; 2-sec-Butyl-4,6-dinitrophenol	88-85-7	8270C
Diphenylamine	122-39-4	8270C
Disulfoton	298-04-4	8270C
Ethyl methanesulfonate	62-50-0	8270C
Famphur	52-85-7	8270C
Fluoranthene	206-44-0	8270C
Fluorene	86-73-7	8270C
Hexachlorobenzene	118-74-1	8270C
Hexachlorobutadiene	87-68-3	8270C
Hexachlorocyclopentadiene	77-47-4	8270C
Hexachloroethane	67-72-1	8270C
Hexachlorophene	70-30-4	8270C
Hexacloroproprene	1888-71-7	8270C
Indeno(1,2,3-cd)pyrene	193-39-5	8270C
Isodrin	465-73-6	8270C
Isophorone	78-59-1	8270C
Isosafrole	120-58-1	8270C
Kepone	143-50-0	8270C
Methapyrilene	91-80-5	8270C
3-Methylcholanthrene	56-49-5	8270C
Methyl methanesulfonate	66-27-3	8270C
2-Methylnaphthalene	91-57-6	8270C
Methyl parathion; Parathion methyl	298-00-0	8270C
Naphthalene	91-20-3	8270C

Appendix IX Constituents	CAS Registry Number	RCRA Method
1,4-Naphthoquinone	130-15-4	8270C
1-Naphthylamine	134-32-7	8270C
2-Naphthylamine	91-59-8	8270C
o-Nitroaniline; 2-Nitroaniline	88-74-4	8270C
m-Nitroaniline;3-Nitroaniline	99-09-2	8270C
p-Nitroaniline; 4-Nitroaniline	100-01-6	8270C
Nitrobenzene	98-95-3	8270C
5-Nitro-o-toluidine	99-55-8	8270C
o-Nitrophenol; 2-Nitrophenol	88-75-5	8270C
p-Nitrophenol; 4-Nitrophenol	100-02-7	8270C
4-Nitroquinoline 1-oxide	56-57-5	8270C
N-nitrosodi-n-butylamine	924-16-3	8270C
N-nitrosodiethylamine	55-18-5	8270C
N-nitrosodimethylamine	62-75-9	8270C
N-nitrosodiphenylamine	86-30-6	8270C
N-nitrosodipropylamine; Di-n-propyl-nitrosamine; N-nitroso-n-dipropylamine	621-64-7	8270C
N-nitrosomethylethylamine	1059-95-6	8270C
N-nitrosomorpholine	59-89-2	8270C
N-nitrosopiperidine	100-75-4	8270C
N-nitrosopyrrolidine	930-55-2	8270C
Parathion	56-38-2	8270C
Pentachlorobenzene	608-93-5	8270C
Pentachloroethane	76-01-7	8270C
Pentachloronitrobenzene	82-68-8	8270C
Pentachlorophenol	87-86-5	8270C
Phenacetin	62-44-2	8270C
Phenanthrene	85-01-8	8270C
Phenol	108-95-2	8270C
p-Phenylenediamine; 1,4-Phenylenediamine	106-50-3	8270C
Phorate	298-02-2	8270C
2-Picoline	109-06-8	8270C
Pronamide	23950-58-5	8270C
Pyrene	129-00-0	8270C
Pyridine	110-86-1	8270C
Safrole	94-59-7	8270C
1,2,4,5-Tetrachlorobenzene	95-94-3	8270C
2,3,4,6-Tetrachlorophenol	58-90-2	8270C
Tetraethylpyrophosphate, Sulfotep	3689-24-5	8270C
o-Toluidine	95-53-4	8270C
1,2,4-Trichlorobenzene	120-82-1	8270C
2,4,5-Trichlorophenol	95-95-4	8270C
2,4,6-Trichlorophenol	88-06-2	8270C
o,o,o-Triethyl phosphoro-thioate; Terbufos	126-68-1	8270C
sym-Trinitrobenzene; 1,3,5-Trinitrobenzene	99-35-4	8270C
ORGANOCHLORINE PESTICIDES/PCBs		
Aldrin	309-00-2	8081A
alpha-BHC; alpha-Benzene hexachloride	319-84-6	8081A

Appendix IX Constituents	CAS Registry Number	RCRA Method
beta-BHC; beta-Benzene hexachloride	309-85-7	8081A
delta-BHC;delta-Benzene hexachloride	319-86-8	8081A
gamma-BHC; gamma-Benzene hexachloride; Lindane	58-89-9	8081A
alpha-Chlordane	5103-71-9	8081A
gamma-Chlordane	5103-74-2	8081A
4,4'-DDD	72-54-8	8081A
4,4'-DDE	72-55-9	8081A
4,4'-DDT	50-29-3	8081A
Dieldrin	60-57-1	8081A
Endosulfan I	959-98-8	8081A
Endosulfan II	33213-65-9	8081A
Endosulfan sulfate	1031-07-8	8081A
Endrin	72-20-8	8081A
Endrin aldehyde	7421-93-4	8081A
Heptachlor	76-44-8	8081A
Heptachlor epoxide	1024-57-3	8081A
Methoxychlor	72-43-5	8081A
Toxaphene	8001-35-2	8081A
Polychlorinated biphenyls; PCBs; Aroclors	1336-36-3, 12674-11-2, 11104-28-2, 11141-16-5, 53469-21-9, 12672-29-6, 11097-69-1, 11096-82-5	8082
HERBICIDES		
2,4-D; 2,4-Dichlorophenoxy-acetic acid	94-75-7	8151A
2,4,5-T; 2,4,5-Trichlorophen-oxyacetic acid	93-76-5	8151A
2,4,5-TP; 2,4,5-Trichloro-phenoxypropanoic acid; Silvex	93-72-1	8151A
METALS		
Antimony	7440-36-0	6010B
Arsenic	7440-38-2	6010B
Barium	7440-39-3	6010B
Beryllium	7440-41-7	6010B
Cadmium	7440-43-9	6010B
Chromium	7440-47-3	6010B
Cobalt	7440-48-4	6010B
Copper	7440-50-8	6010B
Lead	7439-92-1	6010B
Nickel	7440-02-0	6010B
Selenium	7782-49-2	6010B
Silver	7440-22-4	6010B
Thallium	7440-28-0	6010B
Tin	7440-31-5	6010B
Vanadium	7440-62-2	6010B
Zinc	7440-66-6	6010B
Mercury	7439-97-6	7470A

Appendix IX Constituents	CAS Registry Number	RCRA Method
OTHER INORGANICS		
Cyanide	57-12-5	9012B
Sulfide	18496-25-8	9034
DIOXINS		
2,3,7,8-Tetrachlorodibenzo-o-dioxin	1746-01-6	8280A/ 8290
Polychlorinated dibenzo-furans; PCDFs	NA	8280A/ 8290
Polychlorinated dibenzo-p-dioxins; PCDDs	NA	8280A/ 8290

Federal Target Compound and Analyst List

TCL/TAL Constituents	CAS Registry Number	RCRA Method
VOLATILES		
Acetone	67-64-1	8260B
Benzene	71-43-2	8260B
Bromodichloromethane; Dichlorobromomethane	75-27-4	8260B
Bromoform; Tribromomethane	75-25-5	8260B
Carbon disulfide	75-15-0	8260B
Carbon tetrachloride	56-23-5	8260B
Chlorobenzene	108-90-7	8260B
Chloroethane; Ethyl chloride	75-00-3	8260B
Chloroform; Trichlorome-thane	67-66-3	8260B
Dibromochloromethane; Chlorodibromomethane	124-48-1	8260B
o-Dichlorobenzene; 1,2-Dichlorobenzene	95-50-1	8260B
m-Dichlorobenzene; 1,3-Dichlorobenzene	541-73-1	8260B
p-Dichlorobenzene; 1,4-Dichlorobenzene	106-46-7	8260B
1,1-Dichloroethane; Ethylidene chloride	75-34-3	8260B
1,2-Dichloroethane; Ethylene dichloride	107-06-2	8260B
1,1-Dichloroethylene; Vinylidene chloride; 1,1-Dichloroethene	75-35-4	8260B
cis-1,2-Dichloroethylene; cis-1,2-Dichloroethene	156-59-2	8260B
trans-1,2-Dichloroethylene; trans-1,2-Dichloroethene	156-60-5	8260B
1,2-Dichloropropane; Propylene dichloride	78-87-5	8260B
cis-1,3-Dichloropropene	10061-01-5	8260B
trans-1,3-Dichloropropene	10061-02-6	8260B
Ethylbenzene	100-41-4	8260B
2-Hexanone; Methyl butyl ketone	591-78-6	8260B
Methyl bromide; Bromomethane	74-83-9	8260B
Methyl chloride; Chloromethane	74-87-3	8260B
Methyl ethyl ketone; MEK; 2-Butanone	78-93-3	8260B
4-Methyl-2-pentanone; Methyl isobutyl ketone	108-10-1	8260B
Methylene chloride; Dichloromethane	75-09-2	8260B
Styrene	100-42-5	8260B
1,1,2,2-Tetrachloroethane	79-34-5	8260B
Tetrachloroethylene; Tetrachloroethene; Perchloroethylene	127-18-4	8260B
Toluene	108-88-3	8260B
1,1,1-Trichloroethane; Methyl chloroform	71-55-6	8260B
1,1,2-Trichloroethane	79-00-5	8260B
Trichloroethylene; Trichloroethene	79-01-6	8260B
Vinyl chloride; Chloroethene	75-01-4	8260B
Xylene (total)	96-47-6, 108-38-3, 106-42-3, 1330-20-7.	8260B
SEMIVOLATILES		
Acenaphthene	83-32-9	8270C
Acenaphthylene	208-96-8	8270C
Anthracene	120-12-7	8270C
Benzo(a)anthracene; Benzathracene	56-55-3	8270C
Benzo(a)pyrene	50-32-8	8270C

TCL/TAL Constituents	CAS Registry Number	RCRA Method
Benzo(b)fluoranthene	205-99-2	8270C
Benzo(k)fluoranthene	207-08-9	8270C
Benzo(ghi)perylene	191-24-2	8270C
Bis(2-chloroethoxy)methane	111-91-1	8270C
Bis(2-chloroethyl)ether; Dichloroethyl ether	111-44-4	8270C
Bis(2-chloro-1-methylethyl) ether; 2,2-Dichlorodiisopropyl ether; DCIP; 2,2'-Oxybis(1-chloropropane)	108-60-1	8270C
Bis(2-ethylhexyl) phthalate	117-81-7	8270C
4-Bromophenyl phenyl ether	101-55-3	8270C
Butyl benzyl phthalate	85-68-7	8270C
Carbazole	86-74-8	8270C
p-Chloro-m-cresol; 4-Chloro-3-methylphenol	59-50-7	8270C
2-Chloronaphthalene	91-58-7	8270C
2-Chlorophenol	95-57-8	8270C
4-Chlorophenyl phenyl ether	7005-72-3	8270C
Chrysene	218-01-9	8270C
m-Cresol; 3-Methylphenol	108-39-4	8270C
o-Cresol; 2-Methylphenol	95-48-7	8270C
p-Cresol; 4-Methylphenol	106-44-5	8270C
Dibenz(a,h)anthracene	53-70-3	8270C
Dibenzofuran	132-64-9	8270C
Di-n-butyl phthalate	84-74-2	8270C
3,3'-Dichlorobenzidine	91-94-1	8270C
2,4-Dichlorophenol	120-83-2	8270C
Diethyl phthalate	84-66-2	8270C
2,4-Dimethylphenol	105-67-9	8270C
Dimethyl phthalate	131-11-3	8270C
4,6-Dinitro-o-cresol; 4,6-Dinitro-2-methylphenol	534-52-1	8270C
2,4-Dinitrophenol	51-28-5	8270C
2,4-Dinitrotoluene	121-4-2	8270C
2,6-Dinitrotoluene	606-20-2	8270C
Di-n-octyl phthalate	117-84-0	8270C
Fluoranthene	206-44-0	8270C
Fluorene	86-73-7	8270C
Hexachlorobenzene	118-74-1	8270C
Hexachlorobutadiene	87-68-3	8270C
Hexachlorocyclopentadiene	77-47-4	8270C
Hexachloroethane	67-72-1	8270C
Indeno(1,2,3-cd)pyrene	193-39-5	8270C
Isophorone	78-59-1	8270C
2-Methylnaphthalene	91-57-6	8270C
Naphthalene	91-20-3	8270C
o-Nitroaniline; 2-Nitroaniline	88-74-4	8270C
m-Nitroaniline;3-Nitroaniline	99-09-2	8270C
p-Nitroaniline; 4-Nitroaniline	100-01-6	8270C
Nitrobenzene	98-95-3	8270C
o-Nitrophenol; 2-Nitrophenol	88-75-5	8270C
p-Nitrophenol; 4-Nitrophenol	100-02-7	8270C
N-nitrosodiphenylamine	86-30-6	8270C

TCL/TAL Constituents	CAS Registry Number	RCRA Method
N-nitrosodipropylamine; Di-n-propyl-nitrosamine; N-nitroso-n-dipropylamine	621-64-7	8270C
Pentachlorophenol	87-86-5	8270C
Phenanthrene	85-01-8	8270C
Phenol	108-95-2	8270C
Pyrene	129-00-0	8270C
1,2,4-Trichlorobenzene	120-82-1	8270C
2,4,5-Trichlorophenol	95-95-4	8270C
2,4,6-Trichlorophenol	88-06-2	8270C
ORGANOCHLORINE PESTICIDES/PCBs		
Aldrin	309-00-2	8081A
alpha-BHC; alpha-Benzene hexachloride	319-84-6	8081A
beta-BHC; beta-Benzene hexachloride	309-85-7	8081A
delta-BHC;delta-Benzene hexachloride	319-86-8	8081A
gamma-BHC; gamma-Benzene hexachloride; Lindane	58-89-9	8081A
alpha-Chlordane	5103-71-9	8081A
gamma-Chlordane	5103-74-2	8081A
4,4'-DDD	72-54-8	8081A
4,4'-DDE	72-55-9	8081A
4,4'-DDT	50-29-3	8081A
Dieldrin	60-57-1	8081A
Endosulfan I	959-98-8	8081A
Endosulfan II	33213-65-9	8081A
Endosulfan sulfate	1031-07-8	8081A
Endrin	72-20-8	8081A
Endrin aldehyde	7421-93-4	8081A
Endrin ketone	53494-70-5	8081A
Heptachlor	76-44-8	8081A
Heptachlor epoxide	1024-57-3	8081A
Methoxychlor	72-43-5	8081A
Toxaphene	8001-35-2	8081A
Polychlorinated biphenyls; PCBs; Aroclor 1016, 1221, 1232, 1242, 1248, 1254, 1260	12674-11-2, 11104-28-2, 11141-16-5, 53469-21-9, 12672-29-6, 11097-69-1, 11096-82-5	8082
METALS		
Aluminum	7429-90-5	6010B
Antimony	7440-36-0	6010B
Arsenic	7440-38-2	6010B
Barium	7440-39-3	6010B
Beryllium	7440-41-7	6010B
Cadmium	7440-43-9	6010B
Calcium	7440-70-2	6010B
Chromium	7440-47-3	6010B
Cobalt	7440-48-4	6010B
Copper	7440-50-8	6010B

TCL/TAL Constituents	CAS Registry Number	RCRA Method
Iron	7439-89-6	6010B
Lead	7439-92-1	6010B
Magnesium	7439-95-4	6010B
Manganese	7436-96-5	6010B
Nickel	7440-02-0	6010B
Potassium	7440-09-7	6010B
Selenium	7782-49-2	6010B
Silver	7440-22-4	6010B
Sodium	7440-23-5	6010B
Thallium	7440-28-0	6010B
Vanadium	7440-62-2	6010B
Zinc	7440-66-6	6010B
Mercury	7439-97-6	7470A

Federal Priority Pollutant List

PPL Constituents	CAS Registry Number	CWA Method
VOLATILES		
Acrolein	107-02-8	624
Acrylonitrile	107-13-1	624
Benzene	71-43-2	624
Bromodichloromethane; Dichlorobromomethane	75-27-4	624
Bromoform; Tribromomethane	75-25-5	624
Carbon tetrachloride	56-23-5	624
Chlorobenzene	108-90-7	624
Chloroethane; Ethyl chloride	75-00-3	624
2-Chloroethyl vinyl ether	110-75-8	624
Chloroform; Trichloromethane	67-66-3	624
Dibromochloromethane; Chlorodibromomethane	124-48-1	624
o-Dichlorobenzene; 1,2-Dichlorobenzene	95-50-1	624
m-Dichlorobenzene; 1,3-Dichlorobenzene	541-73-1	624
p-Dichlorobenzene; 1,4-Dichlorobenzene	106-46-7	624
1,1-Dichloroethane; Ethylidene chloride	75-34-3	624
1,2-Dichloroethane; Ethylene dichloride	107-06-2	624
1,1-Dichloroethylene; Vinylidene chloride; 1,1-Dichloroethene	75-35-4	624
trans-1,2-Dichloroethylene; trans-1,2-Dichloroethene	156-60-5	624
1,2-Dichloropropane; Propylene dichloride	78-87-5	624
cis-1,3-Dichloropropene	10061-01-5	624
trans-1,3-Dichloropropene	10061-02-6	624
Ethylbenzene	100-41-4	624
Methyl bromide; Bromomethane	74-83-9	624
Methyl chloride; Chloromethane	74-87-3	624
Methylene chloride; Dichloromethane	75-09-2	624
1,1,2,2-Tetrachloroethane	79-34-5	624
Tetrachloroethylene; Tetrachloroethene; Perchloroethylene	127-18-4	624
Toluene	108-88-3	624
1,1,1-Trichloroethane; Methyl chloroform	71-55-6	624
1,1,2-Trichloroethane	79-00-5	624
Trichloroethylene; Trichloroethene	79-01-6	624
Vinyl chloride; Chloroethene	75-01-4	624
SEMIVOLATILES		
Acenaphthene	83-32-9	625
Acenaphthylene	208-96-8	625
Anthracene	120-12-7	625
Benzidine	92-87-5	625
Benzo(a)anthracene; Benzathracene	56-55-3	625
Benzo(a)pyrene	50-32-8	625
Benzo(b)fluoranthene	205-99-2	625
Benzo(k)fluoranthene	207-08-9	625
Benzo(ghi)perylene	191-24-2	625
Bis(2-chloroethoxy)methane	111-91-1	625
Bis(2-chloroethyl)ether; Dichloroethyl ether	111-44-4	625
Bis(2-chloro-1-methylethyl) ether; 2,2-Dichlorodiisopropyl ether; DCIP; 2,2'-Oxybis(1-chloropropane)	108-60-1	625

PPL Constituents	CAS Registry Number	CWA Method
Bis(2-ethylhexyl) phthalate	117-81-7	625
4-Bromophenyl phenyl ether	101-55-3	625
Butyl benzyl phthalate	85-68-7	625
p-Chloro-m-cresol; 4-Chloro-3-methylphenol	59-50-7	625
2-Chloronaphthalene	91-58-7	625
2-Chlorophenol	95-57-8	625
4-Chlorophenyl phenyl ether	7005-72-3	625
Chrysene	218-01-9	625
Dibenz(a,h)anthracene	53-70-3	625
Di-n-butyl phthalate	84-74-2	625
3,3'-Dichlorobenzidine	91-94-1	625
2,4-Dichlorophenol	120-83-2	625
Diethyl phthalate	84-66-2	625
2,4-Dimethylphenol	105-67-9	625
Dimethyl phthalate	131-11-3	625
4,6-Dinitro-o-cresol; 4,6-Dinitro-2-methylphenol	534-52-1	625
2,4-Dinitrophenol	51-28-5	625
2,4-Dinitrotoluene	121-4-2	625
2,6-Dinitrotoluene	606-20-2	625
Di-n-octyl phthalate	117-84-0	625
1,2-Diphenylhydrazine	122-66-7	625
Fluoranthene	206-44-0	625
Fluorene	86-73-7	625
Hexachlorobenzene	118-74-1	625
Hexachlorobutadiene	87-68-3	625
Hexachlorocyclopentadiene	77-47-4	625
Hexachloroethane	67-72-1	625
Indeno(1,2,3-cd)pyrene	193-39-5	625
Isophorone	78-59-1	625
Naphthalene	91-20-3	625
Nitrobenzene	98-95-3	625
o-Nitrophenol; 2-Nitrophenol	88-75-5	625
p-Nitrophenol; 4-Nitrophenol	100-02-7	625
N-nitrosodimethylamine	62-75-9	625
N-nitrosodiphenylamine	86-30-6	625
N-nitrosodipropylamine; Di-n-propyl-nitrosamine; N-nitroso-n-dipropylamine	621-64-7	625
Pentachlorophenol	87-86-5	625
Phenanthrene	85-01-8	625
Phenol	108-95-2	625
Pyrene	129-00-0	625
1,2,4-Trichlorobenzene	120-82-1	625
2,4,6-Trichlorophenol	88-06-2	625
ORGANOCHLORINE PESTICIDES/PCBs		
Aldrin	309-00-2	608
alpha-BHC; alpha-Benzene hexachloride	319-84-6	608
beta-BHC; beta-Benzene hexachloride	309-85-7	608
delta-BHC;delta-Benzene hexachloride	319-86-8	608
gamma-BHC; gamma-Benzene hexachloride; Lindane	58-89-9	608

PPL Constituents	CAS Registry Number	CWA Method
alpha-Chlordane	5103-71-9	608
gamma-Chlordane	5103-74-2	608
4,4'-DDD	72-54-8	608
4,4'-DDE	72-55-9	608
4,4'-DDT	50-29-3	608
Dieldrin	60-57-1	608
Endosulfan I	959-98-8	608
Endosulfan II	33213-65-9	608
Endosulfan sulfate	1031-07-8	608
Endrin	72-20-8	608
Endrin aldehyde	7421-93-4	608
Endrin ketone	53494-70-5	608
Heptachlor	76-44-8	608
Heptachlor epoxide	1024-57-3	608
Methoxychlor	72-43-5	608
Toxaphene	8001-35-2	608
Polychlorinated biphenyls; PCBs; Aroclor 1016, 1221, 1232, 1242, 1248, 1254, 1260	12674-11-2, 11104-28-2, 11141-16-5, 53469-21-9, 12672-29-6, 11097-69-1, 11096-82-5	608
METALS		
Antimony	7440-36-0	200.7
Arsenic	7440-38-2	200.7
Beryllium	7440-41-7	200.7
Cadmium	7440-43-9	200.7
Chromium	7440-47-3	200.7
Copper	7440-50-8	200.7
Lead	7439-92-1	200.7
Nickel	7440-02-0	200.7
Selenium	7782-49-2	200.7
Silver	7440-22-4	200.7
Thallium	7440-28-0	200.7
Zinc	7440-66-6	200.7
Mercury	7439-97-6	245.1
DIOXIN		
2,3,7,8-Tetrachlorodibenzo-p-dioxin (TCDD)	1746-01-6	8280A/ 8290
MISCELLANEOUS		
Asbestos		
Cyanide	57-12-5	SM4500-CN C, E
Phenols	NA	420.1& 420.4

Industrial Hygiene Specific Information

A13.1 TestAmerica Nashville holds accreditation with the American Industrial Hygiene Association for metals, inorganic acids, and particulates analyses. The QA/QC systems and objectives described in this QA Manual are the same as those followed for analysis of industrial hygiene samples. The methods used are those currently approved by the U.S. OSHA and NIOSH, in particular from the NIOSH Manual of Analytical Methods.

A13.2 QUALITY ASSURANCE OBJECTIVES

ANALYTE	LCS ACCURACY %	Reporting Limit µg/FILTER or TUBE
Metals by NIOSH 7303		
Aluminum	80-120	1.25
Arsenic	80-120	1.25
Beryllium	80-120	1.25
Cadmium	80-120	1.25
Calcium	80-120	1.25
Chromium	80-120	2.50
Cobalt	80-120	1.25
Copper	80-120	1.25
Iron	80-120	1.25
Lead	80-120	1.25
Lithium	80-120	1.25
Magnesium	80-120	1.25
Manganese	80-120	1.25
Molybdenum	80-120	1.25
Nickel	80-120	1.25
Selenium	80-120	1.25
Silver	80-120	1.25
Sodium	80-120	1.25
Strontium	80-120	1.25
Thallium	80-120	1.25
Titanium	80-120	1.25
Vanadium	80-120	1.25
Zinc	80-120	12.5
Particulates by NIOSH 0500	NA	NA

NA: Not Applicable

A13.3 SAMPLE HOLDING TIMES AND MEDIA REQUIREMENTS

Parameter	Maximum Holding Time	Cool to 4°C	Sampling Media	Flow Rate, L/min	Maximum Volume, Liters
Metals	Indefinitely	No	MCE Filter Casssette	1-4	200-2000
Particulates	Indefinitely	No	PVC Filter Cassette with Preweighed Filter (Do not use matched-weight filters for paint sampling; use filters pre-weighed by the lab.)	1-2	7-133

A13.4 PROFICIENCY TESTING

A13.4.1 Proficiency testing is performed for each accredited AIHA Field of Testing (FoT). The laboratory participates in and follows all proficiency testing requirements, including frequency of testing, as outlined in current effective AIHA Policy Modules 3, 6A, and 6B.

A13.4.2 Where an AIHA PT program is available for a certified FoT, the laboratory participates in that program. For FoTs not covered by available AIHA supplied PT samples, the laboratory demonstrates competency annually for that FoT through the implementation of one of the following three alternatives outlined in AIHA Policies Module 6A, section 6A.3 and Module 6B, section 6B.3: a third party proficiency testing program, a round robin program between a minimum of 3 AIHA accredited laboratories within the TestAmerica network, or a comprehensive internal QC program.

A13.5 AMENDED REPORTS

A13.5.1 Amended AIHA reports contain the following statement in **bold print** in the comments section on the cover page of the amended report, in addition to the notation requirements in Section 26.9:

> "Supplement to Test Report" along with the WorkOrder number of the original report, the date of the original report, and the time the original report was generated.

A13.6 SUBCONTRACTING

A13.6.1 Unless directed otherwise by a client or regulatory agency, subcontracted work for Fields of Testing covered by the scope of accreditation of TestAmerica Nashville is performed by an AIHA accredited laboratory.

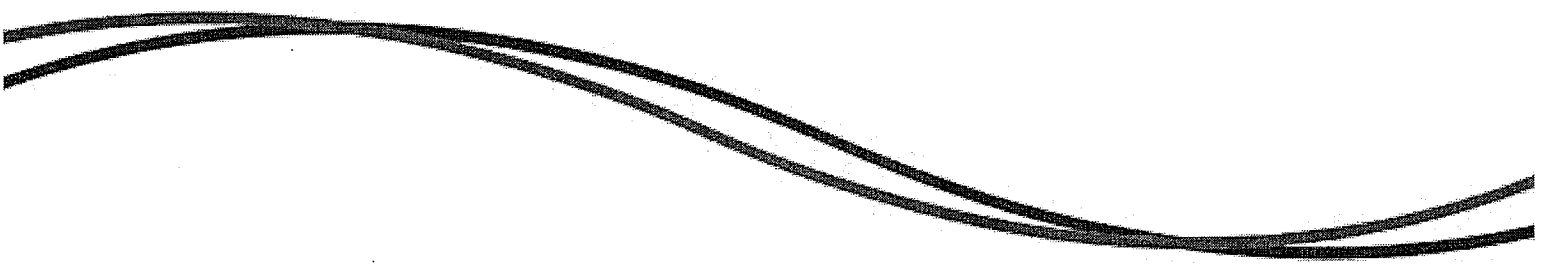
A13.7 TEST REPORTS

A13.7.1 For client projects containing only AIHA-certified parameters (NIOSH methods), "AIHA" is entered into the "Location" field in the Element project set-up screen. If the client's project includes other non-NIOSH methods, the state of origin is entered into the "Location" field to ensure proper reporting per AIHA requirements.

A13.8 Control Limit generation for Duplicate QC data The Taylor* approach is used to generate control limits for Duplicate QC data for NIOSH methods. This is a root-mean-square approach, where the root-mean-square sum of the RPD value is derived and that value is then used as a standard deviation. 2x and 3x that value are plotted as warning and control limits.

*Taylor, John K., Quality Assurance of Chemical Measurement, pp. 22-23, Lewis Publishers, Chelsea, MI 1987.

End of Document





Company Confidential & Proprietary

Cover Page:

Quality Assurance Manual

TestAmerica North Canton
4101 Shuffel Street NW
North Canton, OH 44720
Phone: 330-497-9396
Fax: 330-497-0772

www.testamericainc.com

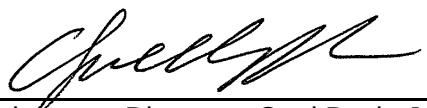
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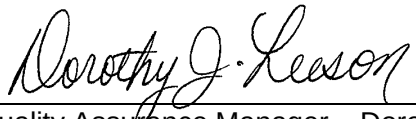
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Quality Assurance Manual
Approval Signatures



Laboratory Director – Opal Davis-Johnson — Date: 07/21/10



Quality Assurance Manager – Dorothy Leeson — Date: 07/21/10



Technical Director – Dr. Mark Bruce — Date: 07/15/10

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CA-L-P-001	Ethics Policy
CA-L-P-002	Contract Compliance Policy
CA-L-S-001	Internal Investigation of Potential Data Discrepancies and Determination for Data Recall
CA-L-S-002	Subcontracting Procedures
CA-Q-S-001	Solvent and Acid Lot Testing and Approval
CA-Q-S-002	Acceptable Manual Integration Practices
CA-Q-S-003	Management of Change Procedure
CA-Q-S-004	Method Compliance & Data Authenticity Audits
CA-Q-S-006	Detection Limits
CA-Q-S-008	Management Systems Review
CA-T-P-001	Qualified Products List
CW-E-M-001	Corporate Environmental Health & Safety Manual
CW-F-P-002	Authorization Matrix
CW-F-P-004	Procurement and Contracts Policy
CW-F-S-007	Controlled Purchases Policy
CW-F-S-018	Vendor Selection
CW-Q-S-001	Corporate Document Control and Archiving
CW-Q-S-002	Writing a Standard Operating Procedure (SOPs)
CA-Q-M-002	Corporate Quality Management Plan
NC-QA-015	Equipment Monitoring and Thermometer Calibration
NC-QA-018	Statistical Evaluation of Data and Development of Control Charts
NC-QA-019	Records Information Management
NC-QA-027	Preparation and Management of Standard Operating Procedures
NC-QA-028	Employee Orientation and Training
NC-QA-029	Nonconformance and Corrective Action System
NC-SC-005	Sample Receiving and Sample Control
NC-SC-006	Sample Procurement Protocol
CA-Q-T-005	Laboratory Documentation
NC-QA-021	Evaluation of Method Detection Limits for Chemical Tests

SECTION 3

INTRODUCTION
(NELAC 5.1 - 5.3)

3.1 INTRODUCTION AND COMPLIANCE REFERENCES

TestAmerica North Canton's Quality Assurance Manual (QAM) is a document prepared to define the overall policies, organization objectives and functional responsibilities for achieving TestAmerica's data quality goals. The laboratory maintains a local perspective in its scope of services and client relations and maintains a national perspective in terms of quality.

The QAM has been prepared to assure compliance with the 2003 National Environmental Laboratory Accreditation Conference (NELAC) standards and ISO/IEC Guide 17025 (2005). In addition, the policies and procedures outlined in this manual are compliant with TestAmerica's Corporate Quality Management Plant, CA-Q-M-002, (CQMP) and the various accreditation and certification programs listed in Appendix 7 (3). The CQMP provides a summary of TestAmerica's quality and data integrity system. It contains requirements and general guidelines under which all TestAmerica facilities shall conduct their operations. The relevant NELAC section is included in the heading of each QAM section.

The QAM has been prepared to be consistent with the requirements of the following documents:

- EPA 600/4-79-019, *Handbook for Analytical Quality Control in Water and Wastewater Laboratories,* EPA, March 1979.
- Test Methods for Evaluating Solid Waste Physical/Chemical Methods (SW846), Third Edition, September 1986, Final Update I, July 1992, Final Update IIA, August 1993, Final Update II, September 1994; Final Update IIB, January 1995; Final Update III, December 1996; Final Update IV, January 2008.
- U.S. Department of Defense, Quality Systems Manual for Environmental Laboratories, Version 4.1, April 2009.
- Federal Register, 40 CFR Parts 136, 141, 172, 173, 178, 179 and 261.
- U.S. Department of Defense, *Quality Systems Manual for Environmental Laboratories*, Final Version 3, January 2006.
- Toxic Substances Control Act (TSCA).

3.2 TERMS AND DEFINITIONS

A Quality Assurance Program is a company-wide system designed to ensure data produced by the laboratory conforms to the standards set by state and/or federal regulations. The program functions at the management level through company goals and management policies, and at the analytical level through Standard Operating Procedures (SOPs) and quality control. The TestAmerica program is designed to minimize systematic error, encourage constructive, documented problem solving, and provide a framework for continuous improvement within the organization.

Refer to Appendix 6 (2) for the Glossary/Acronyms.

3.3 SCOPE / FIELDS OF TESTING

The laboratory analyzes a broad range of environmental and industrial samples every month. Sample matrices vary among effluent water, groundwater, hazardous waste, sludge, wipes, and soils. The Quality Assurance Program contains specific procedures and methods to test samples of differing matrices for chemical, physical and biological parameters. The Program also contains guidelines on maintaining documentation of analytical process, reviewing results, servicing clients and tracking samples through the laboratory. The technical and service requirements of all requests to provide analyses are thoroughly evaluated before commitments are made to accept the work. Measurements are made using published reference methods or methods developed and validated by the laboratory.

The methods covered by this manual include the most frequently requested methodologies needed to provide analytical services in the United States and its territories. The specific list of test methods used by the laboratory can be found in Appendix 4. The approach of this manual is to define the minimum level of quality assurance and quality control necessary to meet requirements. All methods performed by the laboratory shall meet these criteria as appropriate. In some instances, quality assurance project plans (QAPPs), project specific data quality objectives (DQOs) or local regulations may require criteria other than those contained in this manual. In these cases, the laboratory must abide by the requested criteria following review and acceptance of the requirements by the Laboratory Director/Manager , the Quality Assurance (QA) Manager, and the Technical Director. In some cases, QAPPs and DQOs may specify less stringent requirements. The Laboratory Director and the QA Manager must determine if it is in the lab's best interest to follow the less stringent requirements.

- Specific requirements delineated in project plans may supersede general quality requirements described in this manual. Ohio VAP requirements are listed throughout the document.

3.4 MANAGEMENT OF THE MANUAL

3.4.1 Review Process

This manual is reviewed annually by senior laboratory management to assure that it reflects current practices and meets the requirements of the laboratory's clients and regulators as well as the CQMP. Occasionally, the manual may need changes in order to meet new or changing regulations and operations. The QA Manager must review the changes in the normal course of business and incorporate changes into revised sections of the document. All updates must be reviewed by the senior laboratory management staff. The laboratory updates and approves such changes according to our Document Control and Updating Procedures (refer to SOP NC-QA-27 Preparation and Management of SOPs).

SECTION 4

ORGANIZATION AND MANAGEMENT
(NELAC 5.4.1)

4.1 OVERVIEW

TestAmerica North Canton is a local operating unit of TestAmerica Laboratories, Inc. The organizational structure, responsibilities, and authorities of the corporate staff of TestAmerica Laboratories, Inc. are presented in the CQMP. The laboratory has day-to-day independent operational authority overseen by corporate officers (e.g., President, Chief Operating Officer, Corporate Quality Assurance, Corporate Quality Director, etc.). The laboratory operational and support staff work under the direction of the Laboratory Director. The organizational structure for both Corporate and TestAmerica North Canton is presented in Figure 4-1 Employee names are provided to demonstrate range and size of departments however the actual staff members may vary over time. The most current Organization Chart may be obtained from Quality Assurance Manager or Laboratory Director. .

4.2 ROLES AND RESPONSIBILITIES

In order for the Quality Assurance Program to function properly, all members of the staff must clearly understand and meet their individual responsibilities as they relate to the quality program. The following descriptions briefly define each role in its relationship to the Quality Assurance Program. More extensive job descriptions are maintained by laboratory management.

4.2.1 Quality Assurance Program

The responsibility for quality lies with every employee of the laboratory. All employees have access to the QAM, are trained to this manual, and are responsible for knowing the content of this manual and upholding the standards therein. Each person carries out his/her daily tasks in a manner consistent with the goals and in accordance with the procedures in this manual and the laboratory's SOPs. Role descriptions for Corporate personnel are defined in the CQMP. This manual is specific to the operations of TestAmerica North Canton laboratory.

North Canton Laboratory Key Personnel

Name	Position
Rusty Vicinie	General Manager, NE Region
Opal Davis-Johnson	Laboratory Director
Raymond Risden	Operations Manager
Dorothy Leeson	Quality Assurance Manager
Mark Bruce	Technical Director

Rebecca Strait	Client Services Director
Steve Jackson	Regional Safety Director, Waste Management Supervisor
Tammy Tokos	Reporting Group Leader
Olguita Colon	General Chemistry Group Leader
Will Cordell	Field Analytical Group Leader
Steve Earle	Extractions Group Leader
Al Haidet	Shipping Group Leader
Dave Heakin	GC Volatiles Group Leader
Tom Hula	GC/MS Semivolatiles Group Leader
John McFadden	Sample Control Group Leader
Darren Miller	Maintenance Group Leader
Roger Toth	Metals Group Leader
Carolynne Roach	GC Semivolatiles Group Leader
Tom Stiller	GC/MS Volatiles Group Leader
Patrick O'Meara	Project Management Group Leader

4.2.2 Quality Assurance (QA) Manager

The QA Manager has responsibility and authority to ensure the continuous implementation of the quality system

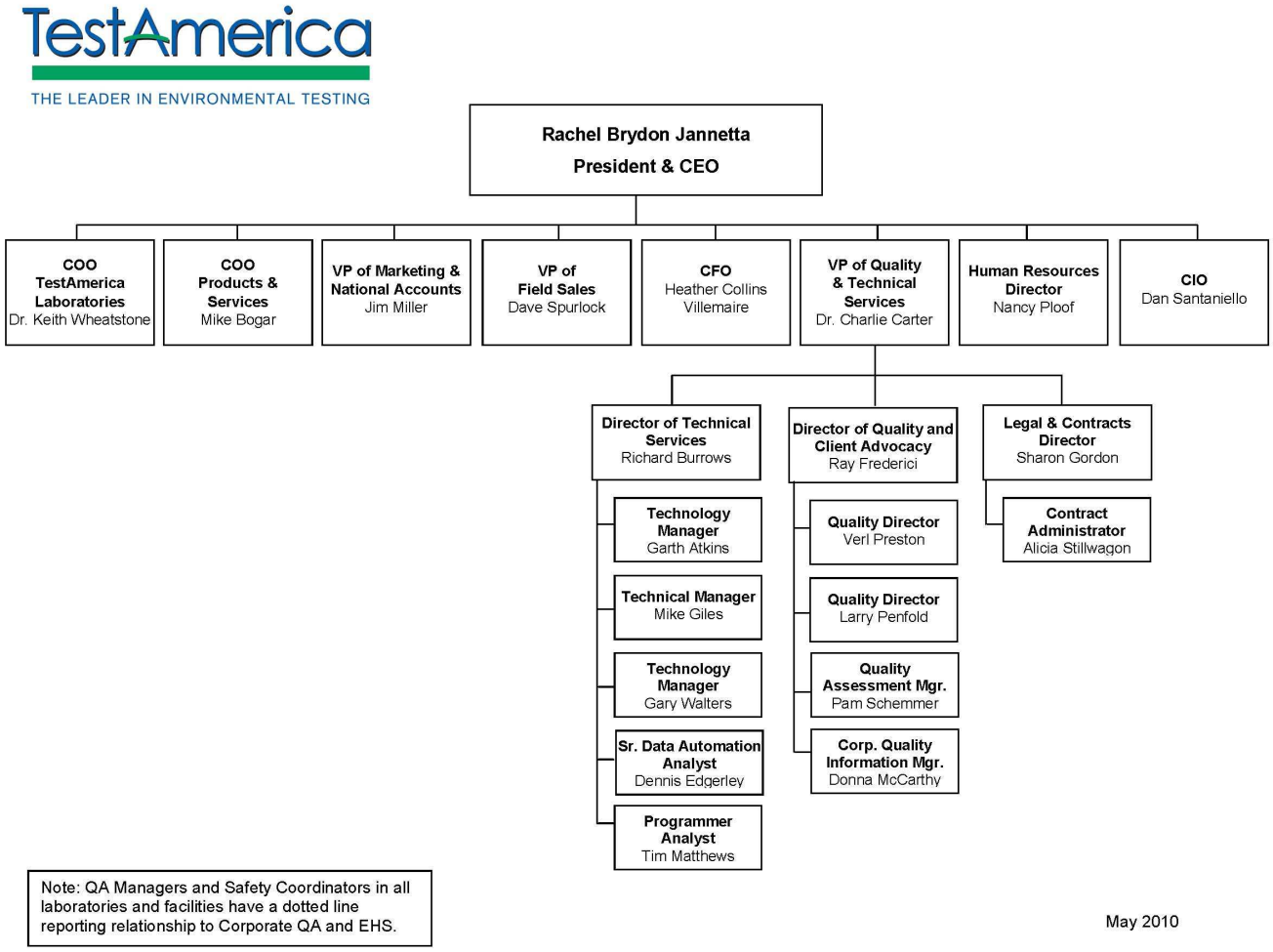
- Ensuring Communication & monitoring standards of performance to ensure that systems are in place to produce the level of quality as defined in this document.
- Notifying laboratory management of deficiencies in the quality system and ensuring corrective action is taken. Procedures that do not meet the standards set forth in the QAM or laboratory SOPs are temporarily suspended following the procedures outlined in Section 12.
- Evaluation of the thoroughness and effectiveness of training.

4.3 DEPUTIES

The following table defines who assumes the responsibilities of key personnel in their absence:

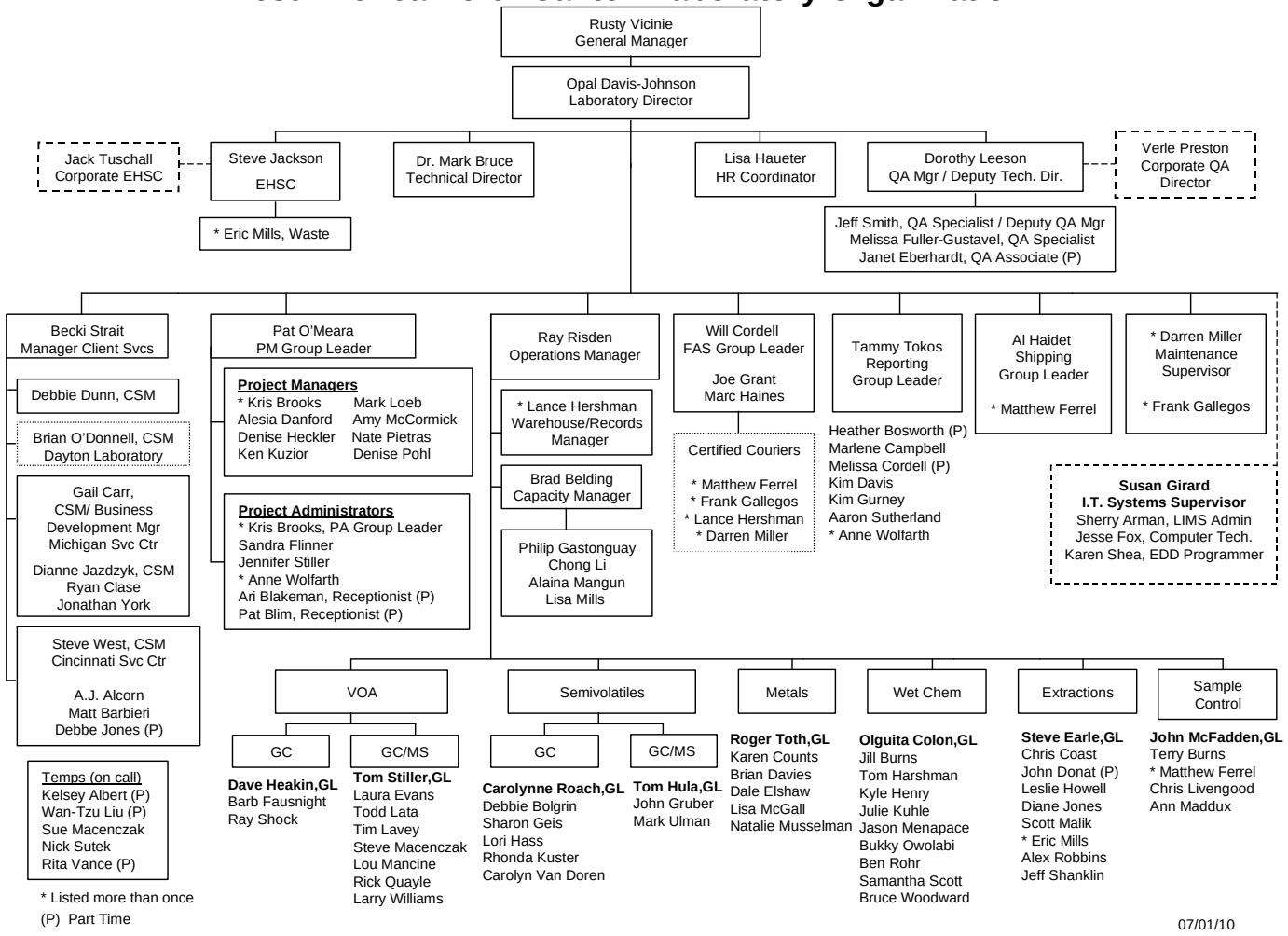
Key Personnel	**Deputy**
Laboratory Director	Technical Director
Quality Assurance Manager	Quality Assurance Specialist
Technical Director	Quality Assurance Manager
EHS Coordinator	Operations Manager

Figure 4-1. Corporate and Laboratory Organization Charts



North Canton Lab Org Chart

TestAmerica North Canton Laboratory Organization



SECTION 5

QUALITY SYSTEM
(NELAC 5.4.2)

5.1 QUALITY POLICY STATEMENT

It is TestAmerica's policy to:

- ❖ Provide data of known qualty to its clients by adhering to approved methodologies, regulatory requirements, and the QA/QC protocols.
- ❖ Effectively manage all aspects of the laboratory and business operations by the highest ethical standards.
- ❖ Continually improve systems and provide support to quality improvement efforts in laboratory, administrative, and managerial activities. TestAmerica recognizes that the implementation of a Quality Assurance program requires management's commitment and support as well as the involvement of the entire staff.
- ❖ Provide clients with the highest level of professionalism and the best service practices in the industry.
- ❖ To comply with the ISO/IEC 17025:2005 International Standard and to continually improve the effectiveness of the management system.

Every staff member at the laboratory plays an integral part in quality assurance and is held responsible and accountable for the quality of their work. It is, therefore, required that all laboratory personnel are trained and agree to comply with applicable procedures and requirements established by this document.

5.2 ETHICS AND DATA INTEGRITY

TestAmerica is committed to ensuring the integrity of its data and meeting the quality needs of its clients. The elements of the TestAmerica Ethics and Data Integrity Program include:

- An Ethics Policy (Corporate Policy CA-L-P-001) and Employee Ethics Statements (Appendix 1)
- Ethics and Compliance Officers (ECOs)
- A training program
- Self-governance through disciplinary action for violations
- A confidential mechanism for anonymously reporting alleged misconduct and a means for conducting internal investigations of all alleged misconduct (Corporate SOP CA-L-S-001)
- Procedures and guidance for recalling data if necessary (Corporate SOP CA-L-S-001)

- Effective external and internal monitoring system that includes procedures for internal audits (Section 16)
- Produce results, which are accurate and include QA/QC information that meets client pre-defined Data Quality Objectives (DQOs).
- Present services in a confidential, honest and forthright manner.
- Provide employees with guidelines and an understanding of the Ethical and Quality Standards of our Industry.
- Operate our facilities in a manner that protects the environment and the health and safety of employees and the public.
- Obey all pertinent federal, state and local laws and regulations and encourage other members of our industry to do the same.
- Educate clients as to the extent and kinds of services available.
- Assert competency only for work for which adequate personnel and equipment are available and for which adequate preparation has been made.
- Promote the status of environmental laboratories, their employees, and the value of services rendered by them.

5.3 QUALITY SYSTEM SUPPORTING DOCUMENTATION

The laboratory's Quality System is communicated through a variety of documents

- Quality Assurance Manual – Each laboratory has a lab-specific Quality Assurance Manual.
- Corporate SOPs and Policies - Corporate SOPs and Policies are developed for use by all relevant laboratories. They are incorporated into the laboratory's normal SOP distribution, training and tracking system. Corporate SOPs may be general or technical.
- Work Instructions - A subset of procedural steps, tasks or forms associated with an operation of a management system, e.g., checklists, preformatted bench sheets, forms.
- Laboratory SOPs – General and technical
- Corporate Quality Policy Memorandums
- Laboratory QA/QC Policy Memorandums

5.3.1 Order of Precedence

In the event of a conflict or discrepancy between policies, the order of precedence is as follows:

- Corporate Quality Policy Memorandum –
- Corporate Quality Management Plan (CQMP)
- Corporate SOPs and Policies
- Laboratory QA/QC Policy Memorandum
- Laboratory Quality Assurance Manual (QAM)
- Laboratory SOPs and Policies

- Other: Work Instructions (WI), memos, flow charts, etc.

Note: The laboratory's has the responsibility and authority to operate in compliance with regulatory requirements of the jurisdiction in which the work is performed. Where the CQMP conflicts with those regulatory requirements, the regulatory requirements of the jurisdiction shall hold primacy. The laboratory's (QAM) shall take precedence over the CQMP in those cases. Any regulatory requirements (e.g.; Ohio VAP, CT RCP, etc) provided in the laboratory specific documents (i.e., QAM and SOPs) take precedence over any policies provided in corporate documents.

5.4 QA/QC OBJECTIVES FOR THE MEASUREMENT OF DATA

Quality Assurance (QA) and Quality Control (QC) are activities undertaken to achieve the goal of producing data that accurately characterize the sites or materials that have been sampled. Quality Assurance is generally understood to be more comprehensive than Quality Control. Quality Assurance can be defined as the integrated system of activities that ensures that a product or service meets defined standards.

Quality Control is generally understood to be limited to the analyses of samples and to be synonymous with the term *"analytical quality control"*. QC refers to the routine application of statistically based procedures to evaluate and control the accuracy of results from analytical measurements. The QC program includes procedures for estimating and controlling precision and bias and for determining reporting limits.

Request for Proposals (RFPs) and Quality Assurance Project Plans (QAPP) provide a mechanism for the client and the laboratory to discuss the data quality objectives in order to ensure that analytical services closely correspond to client needs. The client is responsible for developing the QAPP. In order to ensure the ability of the laboratory to meet the Data Quality Objectives (DQOs) specified in the QAPP, clients are advised to allow time for the laboratory to review the QAPP before being finalized. Additionally, the laboratory must provide support to the client for developing the sections of the QAPP that concern laboratory activities.

Historically, laboratories have described their QC objectives in terms of precision, accuracy, representativeness, comparability, completeness, selectivity and sensitivity (PARCCSS). Equations to derive relevant QC objectives can be found in the method specific SOPs.

5.4.1 Precision

The laboratory objective for precision is to meet the performance for precision demonstrated for the methods on similar samples and to meet data quality objectives of the EPA and/or other regulatory programs. Precision is defined as the degree of reproducibility of measurements under a given set of analytical conditions (exclusive of field sampling variability). Precision is documented on the basis of replicate analysis, usually duplicate or matrix spike (MS) duplicate samples.

5.4.2 Accuracy

The laboratory objective for accuracy is to meet the performance for accuracy demonstrated for the methods on similar samples and to meet data quality objectives of the EPA and/or other

regulatory programs. Accuracy is defined as the degree of bias in a measurement system. Accuracy may be documented through the use of laboratory control samples (LCS) and/or MS. A statement of accuracy is expressed as an interval of acceptance recovery about the mean recovery.

5.4.3 Representativeness

The laboratory objective for representativeness is to provide data which is representative of the sampled medium. Representativeness is defined as the degree to which data represent a characteristic of a population or set of samples and is a measurement of both analytical and field sampling precision. The representativeness of the analytical data is a function of the procedures used in procuring and processing the samples. The representativeness can be documented by the relative percent difference between separately procured, but otherwise identical samples or sample aliquots.

The representativeness of the data from the sampling sites depends on both the sampling procedures and the analytical procedures. The laboratory may provide guidance to the client regarding proper sampling and handling methods in order to assure the integrity of the samples.

5.4.4 Comparability

The comparability objective is to provide analytical data for which the accuracy, precision, representativeness and reporting limit statistics are similar to these quality indicators generated by other laboratories for similar samples, and data generated by the laboratory over time.

The comparability objective is documented by inter-laboratory studies carried out by regulatory agencies or carried out for specific projects or contracts, by comparison of periodically generated statements of accuracy, precision and reporting limits with those of other laboratories.

5.4.5 Completeness

The completeness objective for data is 90% (or as specified by a particular project) expressed as the ratio of the valid data to the total data over the course of the project. Data will be considered valid if they are adequate for their intended use. Data usability must be defined in a QAPP, project scope or regulatory requirement. Data validation is the process for reviewing data to determine its usability and completeness. If the completeness objective is not met, actions will be taken internally and with the data user to improve performance. This may take the form of an audit to evaluate the methodology and procedures as possible sources for the difficulty or may result in a recommendation to use a different method.

5.4.6 Selectivity

Selectivity is defined as the capability of a test method or instrument to respond to a target substance or constituent in the presence of non-target substances. Target analytes are separated from non-target constituents and subsequently identified/detected through one or more of the following, depending on the analytical method: extractions (separation), digestions (separation), inter-element corrections (separation), use of matrix modifiers (separation),

specific retention times (separation and identification), confirmations with different columns or detectors (separation and identification), specific wavelengths (identification), specific mass spectra (identification), specific electrodes (separation and identification), etc.

5.4.7 Sensitivity

Sensitivity refers to the amount of analyte necessary to produce a detector response that can be reliably detected (Method Detection Limit) or quantified (Reporting Limit).

5.5 CRITERIA FOR QUALITY INDICATORS

The laboratory maintains a Control Limit Report that summarizes the precision and accuracy acceptability limits for performed analyses. This summary includes an effective date, is updated each time new limits are generated, and are managed by the laboratory's QA Department. Unless otherwise noted, limits within these tables are laboratory generated. Some acceptability limits are derived from US EPA methods when they are required. Where U.S. EPA method limits are not required, the laboratory has developed limits from evaluation of data from similar matrices. Criteria for development of control limits are contained in NC-QA-018 Statistical Evaluation of Data and Development of Control Charts and in Section 24).

5.6 STATISTICAL QUALITY CONTROL

Statistically-derived precision and accuracy limits are required by selected methods (such as SW-846) and programs The laboratory routinely utilizes statistically-derived limits to evaluate method performance and determine when corrective action is appropriate. The analysts are instructed to use the current limits in the laboratory (dated and approved by the Group Leader and QA Manager) and entered into the Laboratory Information Management System (LIMS). The Quality Assurance Department maintains an archive of all limits used within the laboratory. If a method defines the QC limits, the method limits are used.

If a method requires the generation of historical limits, the lab develops such limits from recent data in the QC database of the LIMS following the guidelines described in Section 25. All calculations and limits are documented and dated when approved and effective. On occasion, a client requests contract-specified limits for a specific project.

Surrogate recoveries are determined for a specific time period as defined above. The resulting ranges are entered in LIMS.

Current QC limits are entered and maintained in the LIMS analyte database. As sample results and the related QC are entered into LIMS, the sample QC values are compared with the limits in LIMS to determine if they are within the acceptable range. The analyst then evaluates if the sample needs to be rerun or re-extracted/rerun or if a comment should be added to the report explaining the reason for the QC outlier.

5.6.1 QC Charts

In-house limits for all QC data must be evaluated and compared to the limits published in the methods for applicable matrices. Method limits must be employed until sufficient QC data are acquired. A minimum of 20 to 30 data points are recommended to establish the in-house QC limits. Calculated results of the QC (LCS) samples are evaluated by comparing against control limits (3-sigma).

Control charts are used to develop control limits, trouble-shoot analytical problems, and, in conjunction with the non-conformance system, to monitor for trends. Program-specific data analysis requirements for control charts are followed as required for data generated under those programs. These additional requirements shall be documented in a QAPP or QAS.

5.7 QUALITY SYSTEM METRICS

In addition to the QC parameters discussed above, the entire Quality System is evaluated on a monthly basis through the use of specific metrics (refer to Section 16). These metrics are used to drive continuous improvement in the laboratory's Quality System.

SECTION 6

DOCUMENT CONTROL
(NELAC 5.4.3)

6.1 OVERVIEW

The QA Department is responsible for the control of documents used in the laboratory to ensure that approved, up-to-date documents are in circulation and out-of-date (obsolete) documents are archived or destroyed. The following documents, at a minimum, must be controlled at each laboratory Facility:

- Laboratory Quality Assurance Manual
- Laboratory Standard Operating Procedures (SOP)
- Laboratory Policies
- Work Instructions and Forms
- Corporate Policies and Procedures distributed outside the intranet

Corporate Quality posts Corporate Manuals, SOPs, Policies, Work Instructions, White Papers, and Training Materials on the company intranet site. These Corporate documents are only considered controlled when they are read on the company intranet site. Printed copies are considered uncontrolled unless the laboratory physically distributes them as controlled documents. A detailed description of the procedure for issuing, authorizing, controlling, distributing, and archiving Corporate documents is found in Corporate SOP CW-Q-S-001, Corporate Document Control and Archiving. The laboratory's internal document control procedure is defined in SOP NC-QA-027, Preparation and Management of Standard Operataing Procedures.

The laboratory QA Department also maintains access to various references and document sources integral to the operation of the laboratory. This includes reference methods and regulations. The laboratory also maintains instrument manuals (hard or electronic copies).

The laboratory maintains control of records for raw analytical data and supporting records such as audit reports and responses, logbooks, standard logs, training files, MDL studies, Proficiency Testing (PT) studies, certifications and related correspondence, and Corrective Action reports. Raw analytical data consists of bound logbooks, instrument printouts, any other notes, magnetic media, electronic data and final reports.

6.2 DOCUMENT APPROVAL AND ISSUE

The pertinent elements of a document control system for each document include a unique document title and number, the number of pages of the item, the effective date, revision number, and the laboratory name. The QA Department is responsible for the maintenance of this system.

Controlled documents are authorized by the QA Department and other management. In order to develop a new document, a staff member submits an electronic draft to the QA Department

for suggestions and approval before use. Upon approval, QA personnel add the identifying version information to the document, and retains the official document on file. The official document is provided to all applicable operational units (may include electronic access). Controlled documents are identified as such and records of their distribution are kept by the QA Department. Document control may be achieved by either electronic or hardcopy distribution.

The QA Department maintains a list of the official versions of controlled documents.

Quality System Policies and Procedures must be reviewed at a minimum of every 24 months, and revised as appropriate. For procedures associated with DoD and Ohio VAP project work, applicable SOPs and Policies are reviewed every 12 months. Changes to documents occur when a procedural change warrants.

6.3 PROCEDURES FOR DOCUMENT CONTROL POLICY

For changes to the QA Manual, refer to SOPs NC-QA-019 and CW-Q-S-001. Uncontrolled copies must not be used within the laboratory. Previous revisions and back-up data are stored by the QA/QC Department.

For changes to SOPs, refer to Corporate SOP CW-Q-S-002, Writing a Standard Operating Procedure (SOP), and SOP NC-QA-027, Preparation and Management of Standard Operating Procedures.

Forms, worksheets, Work Instructions, and information are organized by department in the QA office. Electronic versions are kept on a hard drive in the QA department; hard copies are kept in QA files. The procedure for the care of these documents is in SOP NC-QA-027.

6.4 OBSOLETE DOCUMENTS

All invalid or obsolete documents are removed, or otherwise prevented from unintended use. The laboratory has specific procedures as described above to accomplish this. In general, obsolete documents are collected from employees according to distribution lists and are marked obsolete on the cover or destroyed. At least one copy of the obsolete document is archived according to SOP NC-QA-027.

SECTION 7

SERVICE TO THE CLIENT
(NELAC 5.4.7)

7.1 OVERVIEW

The laboratory has established procedures for the review of work requests and contracts, oral or written. The procedures include evaluation of the laboratory's capability and resources to meet the contract's requirements within the requested time period. All requirements, including the methods to be used, must be adequately defined, documented and understood. For many environmental sampling and analysis programs, testing design is site or program specific and does not necessarily "fit" into a standard laboratory service or product. It is the laboratory's intent to provide both standard and customized environmental laboratory services to our clients.

A thorough review of technical and QC requirements contained in contracts is performed to ensure project success. The appropriateness of requested methods, and the lab's capability to perform them must be established. Projects, proposals and contracts are reviewed for adequately defined requirements and the laboratory's capability to meet those requirements. Alternate test methods that are capable of meeting the clients' requirements may be proposed by the lab. A review of the lab's capability to analyze non-routine analytes is also part of this review process.

All projects, proposals and contracts are reviewed for the client's requirements in terms of compound lists, test methodology requested, sensitivity (detection and reporting levels), accuracy, and precision requirements (Percent Recovery and RPD). The reviewer ensures that the laboratory's test methods are suitable to achieve these regulatory and client requirements and that the laboratory holds the appropriate certifications and approvals to perform the work. The laboratory and any potential subcontract laboratories must be certified, as required, for all proposed tests.

The laboratory must determine if it has the necessary physical, personnel and information resources to meet the contract, and if the personnel have the expertise needed to perform the testing requested. Each proposal is checked for its impact on the capacity of the laboratory's equipment and personnel. As part of the review, the proposed turnaround time must be checked for feasibility.

Electronic or hard-copy deliverable requirements are evaluated against the laboratory's capacity for production of the documentation.

If the laboratory cannot provide all services but intends to subcontract such services, whether to another TestAmerica facility or to an outside firm, this must be documented and discussed with the client prior to contract approval (refer to Section 8 for Subcontracting Procedures).

The laboratory informs the client of the results of the review if it indicates any potential conflict, deficiency, lack of accreditation, or inability of the lab to complete the work satisfactorily. Any discrepancy between the client's requirements and the laboratory's capability to meet those requirements is resolved in writing before acceptance of the contract. It is necessary that the

contract be acceptable to both the laboratory and the client. Amendments initiated by the client and/or TestAmerica, are documented in writing.

All contracts, QAPPs, Sampling and Analysis Plans (SAPs), contract amendments, and documented communications become part of the project record.

The same contract review process used for the initial review is repeated when there are amendments to the original contract by the client, and the participating personnel are informed of the changes.

7.2 REVIEW SEQUENCE AND KEY PERSONNEL

Appropriate personnel must review the work request at each stage of evaluation.

For routine projects and other simple tasks, a review by the Project Manager (PM) is considered adequate. The PM confirms that the laboratory has any required certifications, that it can meet the clients' data quality and reporting requirements and that the lab has the capacity to meet the clients turn around needs. It is recommended that, where there is a sales person assigned to the account, an attempt should be made to contact that sales person to inform them of the incoming samples.

For new, complex or large projects, the opportunity is forwarded to a Customer Service Manager (CSM) for review. The CSM contacts the appropriate Sales Executive (National Account Manager, Key Account Executive, Regional Account Executive, and/or Program Manager) to determine which lab will receive the work based on the scope of work and other requirements, including certification, testing methodology, reporting specifications, and available capacity to perform the work. The contract review process is outlined in TestAmerica's Corporate SOP CA-L-P-002**,** Contract Compliance Policy.

This review encompasses all facets of the operation. The scope of work is distributed to the appropriate personnel, as needed based on scope of contract, to evaluate all of the requirements shown above (not necessarily in the order below):

- Legal & Contracts Director
- Laboratory Customer Service Manager
- Laboratory Operations Manager
- Laboratory and/or Corporate Technical Director
- Laboratory and/or Corporate Information Technology Managers/Directors
- Regional and/or National Account representatives
- Laboratory and/or Corporate Quality Assurance Managers
- Laboratory and/or Corporate Environmental Health and Safety Managers/Directors
- The Laboratory Director/Manager reviews the formal laboratory quote, and makes final acceptance for their facility.
- Based on the level of discount extended for the project, approval of the General Manager or Sales Director may also be required.

The Customer Service Manager or local Account Executive then submits the final proposal to the client.

In the event that one of the above personnel is not available to review the contract, his or her backup will fulfill the review requirements.

The Legal & Contracts Director (or their designee) maintains copies of all signed contracts. The Laboratory Director also maintains an electronic copy of any contract signed at the local level.

7.3 DOCUMENTATION

Appropriate records are maintained for every contract or work request. All stages of the contract review process are documented and include records of any significant changes. Documents are reviewed by the Laboratory Director and stored on the laboratory's public drive.

The contract must be distributed to and maintained by the Corporate Contracts Department and the applicable Account Executive. A copy of the contract must be filed electronically by the Laboratory Director. Quotes must be archived electronically in the laboratory quote module (TALs) or in the public shared drive if an off-TALs quote is submitted.

Records are maintained of pertinent discussions with a client relating to the client's requirements or the results of the work during the period of execution of the contract. The PM keeps email records or a phone log of conversations with the client.

7.3.1 Project-Specific Quality Planning

Communication of contract-specific technical and QC criteria is an essential activity in ensuring the success of site specific testing programs. To achieve this goal, the laboratory assigns a PM to each client. The PM is the first point of contact for the client. It is the PM's responsibility to ensure that project specific technical and QC requirements are effectively evaluated and communicated to the laboratory personnel before and during the project. QA department involvement may be needed to assist in the evaluation of custom QC requirements.

PM's are the primary client contact and they ensure resources are available to meet project requirements. Although PM's do not have direct reports or staff in production, they coordinate opportunities and work with laboratory management and supervisory staff to ensure available resources are sufficient to perform work for the client's project. Project management is positioned between the client and laboratory resources.

Prior to work on a new project, the dissemination of project information and/or project opening meetings may occur to discuss schedules and unique aspects of the project. Items to be discussed may include the project technical profile, turnaround times, holding times, methods, analyte lists, reporting limits, deliverables, sample hazards, or other special requirements. The PM introduces new projects to the laboratory staff through project kick-off meetings or to the supervisory staff during production meetings. These meetings provide direction to the laboratory staff in order to maximize production and client satisfaction, while maintaining quality. In addition,

project notes may be associated with each sample batch as a reminder upon sample receipt and analytical processing.

During the project, any change that may occur within an active project is agreed upon between the client/regulatory agency and the PM/laboratory. These changes, e.g., use of a non-standard method or modification of a method, and approvals must be documented prior to implementation. Documentation pertains to any document, e.g., letter, e-mail, variance, contract addendum, which has been signed by both parties.

Such changes are also communicated to the laboratory. Project-specific changes made after samples are in-house are communicated through Change Order forms.

Programmatic and/or method changes are communicated via email transmittal and/or in meetings with the applicable Operations Managers. If the modification includes use of a non-standard method, or significant modification of a method, documentation of the modification is made in the case narrative of the applicable data report(s).

The laboratory strongly encourages client visits to the laboratory and for formal/informal information sharing session with employees in order to effectively communicate ongoing client needs as well as project specific details for customized testing programs.

7.4 SPECIAL SERVICES

The laboratory cooperates with clients and their representatives to monitor the laboratory's performance in relation to work performed for the client. It is the laboratory's goal to meet all client requirements in addition to statutory and regulatory requirements. The laboratory has procedures to ensure confidentiality to clients (Section 15 and 25).

Note: ISO 17025/NELAC 2003 states that a laboratory "shall afford clients or their representatives cooperation to clarify the client's request". This topic is discussed in Section 7.

The laboratory's standard procedures for reporting data are described in Section 25. Special services are also available and provided upon request. These services include:

- Reasonable access for our clients or their representatives to the relevant areas of the laboratory for the witnessing of tests performed for the client.
- Assist client-specified third-party data validators as specified in the client's contract.
- Supplemental information pertaining to the analysis of their samples. Note: An additional charge may apply for additional data/information that was not requested prior to the time of sample analysis or previously agreed upon.

7.5 CLIENT COMMUNICATION

Customer Service Managers (CSMs) and Project Managers (PMs) are the primary communication link to the clients. They must inform their clients of any delays in project completion as well as any non-conformances in either sample receipt or sample analysis. Project Management must maintain ongoing client communication throughout the entire client project.

Technical Directors, Operation Manager, or Group Leaders are available to discuss any technical questions or concerns the client may have.

7.6 REPORTING

The laboratory works with our clients to produce any special communication reports required by the contract.

7.7 CLIENT SURVEYS

The laboratory assesses both positive and negative client feedback. The results are used to improve overall laboratory quality and client service.

TestAmerica Sales and Marketing teams periodically develop lab and client-specific surveys to assess client satisfaction.

SECTION 8

SUBCONTRACTING OF TESTS
(NELAC 5.4.5)

8.1 OVERVIEW

For the purpose of this quality manual, the phrase subcontract laboratory refers to a laboratory external to theTestAmerica Laboratories. The phrase “work sharing” refers to internal transfers of samples between the TestAmerica Laboratories. The term “outsourcing” refers to the act of subcontracting tests.

When contracting with our clients, the laboratory makes commitments regarding the services to be performed and the data quality for the results to be generated. When the need arises to outsource testing for our clients because project scope, changes in laboratory capabilities, capacity or unforeseen circumstances, we must be assured that the subcontractors or work sharing laboratories understand the requirements and must meet the same commitments we have made to the client. Refer to TestAmerica’s Corporate SOPs on Subcontracting Procedures (CA-L-S-002) and the Work Sharing Process SOP (CA-C-S-001).

When outsourcing analytical services, the laboratory must assure, to the extent necessary, that the subcontract or work sharing laboratory maintains a program consistent with the requirements of this document, the requirements specified in NELAC/ISO 17025 and/or the client’s Quality Assurance Project Plan (QAPP). All QC guidelines specific to the client’s analytical program are transmitted to the subcontractor and agreed upon before sending the samples to the subcontract facility. Additionally, work requiring accreditation must be placed with an appropriately accredited laboratory. The laboratory performing the subcontracted work must be identified in the final report, as must non-NELAC accredited work, where required.

For DOD projects, the subcontractor laboratories used must have an established and documented laboratory quality system that complies with DoD QSM requirements. The subcontractor laboratories are evaluated following the procedures outlined below and as seen in Figure 8-2. The subcontractor laboratory must receive project-specific approval from the DoD client before any samples are analyzed.

The QSM has five specific requirements for subcontracting:

1. Subcontractor laboratories must have an established laboratory quality system that complies with the QSM.

2. Subcontractor laboratories must be approved by the specific DoD component laboratory approval process.

3. Subcontractor laboratories must demonstrate the ability to generate acceptable results from the analysis of PT samples, subject to availability, using each applicable method, in the specified matrix, and provide appropriate documentation to the DoD client.

4. Subcontractor laboratories must receive project-specific approval from the DoD client before any samples are analyzed.

5. Subcontractor laboratories are subject to project-specific, on-site assessments by the DoD client or their designated representatives.

Project Managers (PMs) or Customer Service Managers (CSM) for the Export Lab are responsible for obtaining client approval prior to outsourcing any samples. The laboratory must advise the client of a subcontract or work sharing arrangement in writing and when possible approval from the client must be retained in the project folder.

Note: In addition to the client, some regulating agencies, such as the US Army Corps of Engineers and the USDA, require notification prior to placing such work.

8.2 QUALIFYING AND MONITORING SUBCONTRACTORS

Whenever a PM or Customer Service Manager (CSM) becomes aware of a client requirement or laboratory need where samples must be outsourced to another laboratory, the other laboratory(s) shall be selected based on the following:

- The first priority is to attempt to place the work in a qualified TestAmerica laboratory
- Firms specified by the client for the task. (Documentation that a subcontractor was designated by the client must be maintained with the project file. This documentation can be as simple as placing a copy of an e-mail from the client in the project folder.)
- Firms listed as pre-qualified and currently under a subcontract with TestAmerica. A listing of all approved subcontracting laboratories and supporting documentation is available on the TestAmerica intranet site. Verify necessary accreditation, where applicable (e.g., on the subcontractors NELAC, A2LAA accreditation or State Certification.
- Firms identified in accordance with the company's Small Business Subcontracting program as small, women-owned, veteran-owned and/or minority-owned businesses
- NELAC or A2LA-accredited laboratories
- In addition, the firm must hold the appropriate certification to perform the work required

All TestAmerica Laboratories are pre-qualified for work sharing, provided they hold the appropriate accreditations, can adhere to the project/program requirements, and the client approved sending samples to that laboratory. The client must provide acknowledgement that the samples can be sent to that facility (an e-mail is sufficient documentation or if acknowledgement is verbal, the date, time, and name of person providing acknowledgement must be documented). The originating laboratory is responsible for communicating all technical, quality, and deliverable requirements as well as other contract needs. Refer to Corporate SOP CA-C-S-001, Work Sharing Process.

When the potential subcontract laboratory has not been previously approved, CSMs or PMs may nominate a laboratory as a subcontractor based on need. The decision to nominate a laboratory must be approved by the Laboratory Director/Manager. The Laboratory Director/Manager requests that the QA Manager begin the process of approving the subcontract

laboratory as outlined in Corporate SOP CA-L-S-002, Subcontracting Procedures. The client must provide acknowledgement that the samples can be sent to that facility. (An e-mail is sufficient documentation; or if acknowledgement is verbal, the date, time, and name of person providing acknowledgement must be documented.)

8.2.1 Once the appropriate accreditation and legal information is received by the laboratory, it is evaluated for acceptability (where applicable) and forwarded to Corporate Contracts for formal contracting with the laboratory. They must add the lab to the approved list on the intranet site, along with the associated documentation and notify the Finance Group for J.D.Edwards.

8.2.2 The client must assume responsibility for the quality of the data generated from the use of a subcontractor they have requested the lab to use. The qualified subcontractors on the intranet site are to meet minimal standards. TestAmerica does not certify laboratories. The subcontractor is on our approved list, and can only be recommended to the extent that we would use them.

8.2.3 The status and performance of qualified subcontractors must be monitored periodically by the Corporate Contracts and/or Quality Departments. Any problems identified must be brought tothe attention of TestAmerica's Corporate Finance or Corporate Quality personnel.

- Complaints must be investigated. Documentation of the complaint, investigation, and corrective action must be maintained in the subcontractor file on the intranet site. Complaints are posted using the Vendor Performance Report**.**
- Information must be updated on the intranet when new information is received from the subcontracted laboratories.
- Subcontractors in good standing must be retained on the intranet listing. The QA Manager must notify all TestAmerica laboratories, Corporate Quality, and Corporate Contracts if any laboratory requires removal from the intranet site. This notification must be posted on the intranet site and e-mailed to all Lab Directors/Managers, QA Managers, and Sales Personnel.

8.3 OVERSIGHT AND REPORTING

The CSM or PM must request that the selected subcontractor be presented with a subcontract, if one is not already executed between the laboratory and the subcontractor. The subcontract must include terms which flow down the requirements of our clients, either in the subcontract itself or through the mechanism of work orders relating to individual projects. A standard subcontract and the Lab Subcontractor Vendor Package (posted on the intranet) can be used to accomplish this, and the Legal & Contracts Director can tailor the document or assist with negotiations, if needed. The PM or CSM responsible for the project must advise and obtain client consent to the subcontract as appropriate, and provide the scope of work to ensure that the proper requirements are made a part of the subcontract and are made known to the subcontractor.

Prior to sending samples to the subcontracted laboratory, the PM confirms their certification status to determine if it's current and scope-inclusive. For TestAmerica Laboratories, certifications can be viewed on the company's TotalAccess Database.

The Sample Control Department is responsible for ensuring compliance with QA requirements and applicable shipping regulations when shipping samples to a subcontracted laboratory.

All subcontracted samples must be accompanied by a Chain of Custody (COC). A copy of the original COC sent by the client must be included with all samples subbed within the TestAmerica.

Through communication with the subcontracted laboratory, the PM monitors the status of the subcontracted analyses, facilitates successful execution of the work, and ensures the timeliness and completeness of the analytical report.

Non-NELAC accredited work must be identified in the subcontractor's report as appropriate. If NELAC accreditation is not required, the report does not need to include this information.

Reports submitted from subcontractor laboratories are not altered and are included in their original form in the final project report. This clearly identifies the data as being produced by a subcontractor facility. If subcontract laboratory data is incorporated into the laboratory EDD, i.e., imported, the report must explicitly indicate the specific lab that produced the data and identify the specific methods and samples.

Note: The results submitted by a TestAmerica work-sharing laboratory may be transferred electronically and the results reported by the TestAmerica work-sharing lab are identified on the final report. The report must explicitly indicate which lab produced the data for which methods and samples. The final report must include a copy of the completed COC for all work sharing reports.

8.4 CONTINGENCY PLANNING

The Laboratory Director/Manager may waive the full qualification of a subcontractor process temporarily to meet emergency needs. In the event this provision is utilized, the QA Manager will be required to verify certifications. The comprehensive approval process must then be initiated within 30 calendar days of subcontracting.

Figure 8-1. Example - Client-Approved Subcontractor Form

Client Information:

Client Name & Account Number:_____

Client Contact: _____

Client Address: _____

_____

Project Information: (Please choose all applicable.)

- **Certification required: □ State □ NELAC □ A2LA □ Method___**

  □ Target compound_____ □ Other_____ □ N/A

- **Required Turn around time (method provisional)**_____

Subcontractor's Information:

Subcontractor's Name: _____

Subcontractor's Contact: _____

Subcontractor's Email: _____

Subcontractor's Address: _____

_____

Subcontractor's Phone Number: _____

Analytical Test/Compound/Method to be subcontracted: _____

Certification Statement:

I hereby give ***[Insert Lab Name]*** permission to use the above noted subcontractor for the above noted testing procedures/methods. I realize that the above subcontractor will be held liable for the validity of the above mentioned testing procedures/methods. All subcontractors shall meet the requirements as spelled out in project information and will follow all analytical holding times and turn around times for analytical reports. The subcontract laboratory, and not TestAmerica, will be held liable for liquidated damages for delays in subcontracted analytical reports and/or electronic data deliverables.

Client Signature **Date**

SECTION 9

PURCHASING SERVICES AND SUPPLIES
(NELAC 5.4.6)

9.1 OVERVIEW

Evaluation and selection of suppliers and vendors is performed, in part, on the basis of the quality of their products, their ability to meet the demand for their products on a continuous and short term basis, the overall quality of their services, their past history, and competitive pricing. This is achieved through evaluation of objective evidence of quality furnished by the supplier, which can include certificates of analysis, recommendations, and proof of historical compliance with similar programs for other clients. To ensure that quality critical consumables and equipment conform to specified requirements, which may affect quality, all purchases from specific vendors are approved by a member of the supervisory or management staff. Capital expenditures are made in accordance with the TestAmerica's Corporate Controlled Purchases Procedure, SOP CW-F-S-007.

Contracts must be signed in accordance with TestAmerica's Corporate Authorization Matrix Policy, Policy CW-F-P-002. Request for Proposals (RFP's) must be issued where more information is required from the potential vendors than just price. Process details are available in TestAmerica's Corporate Procurement and Contracts Policy (Policy CW-F-P-004). RFP's allow TestAmerica to determine if a vendor is capable of meeting requirements such as supplying all of the TestAmerica facilities, meeting required quality standards and adhering to necessary ethical and environmental standards. The RFP process also allows potential vendors to outline any additional capabilities they may offer.

9.2 GLASSWARE

Glassware used for volumetric measurements must be Class A or verified for accuracy according to laboratory procedure. Pyrex (or equivalent) glass must be used where possible. For safety purposes, thick-wall glassware must be used where available.

9.3 REAGENTS, STANDARDS & SUPPLIES

Purchasing guidelines for equipment and reagents must meet the requirements of the specific method and testing procedures for which they are being purchased. Solvents and acids are pre-tested in accordance with TestAmerica's Corporate SOP on Solvent and Acid Lot Testing and Approval, SOP CA-Q-S-001.

9.3.1 Purchasing

Chemical reagents, solvents, glassware, and general supplies are ordered as needed to maintain sufficient quantities on hand. Materials used in the analytical process must be of a known quality. The wide variety of materials and reagents available makes it advisable to specify recommendations for the name, brand, and grade of materials to be used in any

determination. This information is contained in the method SOP. The analyst may check the item out of the on-site consignment system that contains items approved for laboratory use

9.3.2 Receiving

It is the responsibility of the Warehouse Manager to receive the shipment. It is the responsibility of the analyst who ordered the materials to date the material when received. Once the ordered reagents or materials are received, the analyst compares the information on the label or packaging to the original order to ensure that the purchase meets the quality level specified. Material Safety Data Sheets (MSDSs) are kept on a backup disc located in the Wet Chemistry bullpen and available online through the Company's intranet website. Anyone may review these for relevant information on the safe handling and emergency precautions of on-site chemicals.

9.3.3 Specifications

All methods in use in the laboratory specify the grade of reagent that must be used in the procedure. If the quality of the reagent is not specified, it may be assumed that it is not significant in that procedure and, therefore, any grade reagent may be used. It is the responsibility of the analyst to check the procedure carefully for the suitability of grade of reagent. Specifications are listed in SOP NC-QA-017, Reagents and Standards.

Chemicals must not be used past the manufacturer's expiration date and must not be used past the expiration time noted in a method SOP. If expiration dates are not provided, the laboratory may contact the manufacturer to determine an expiration date.

The laboratory assumes a five-year expiration date on inorganic dry chemicals unless noted otherwise by the manufacturer or by the reference source method. Chemicals must not be used past themanufacturer's or SOPs expiration date unless "verified" (refer to Item 3 listed below).

- An expiration date cannot be extended if the dry chemical is discolored or appears otherwise physically degraded, the dry chemical must be discarded.

- Expiration dates can be extended if the dry chemical is found to be satisfactory based on acceptable performance of quality control samples (Continuing Calibration Verification (CCV), Blanks, Laboratory Control Sample (LCS), etc.).

- If the dry chemical is used for the preparation of standards, the expiration dates can be extended six months if the dry chemical is compared to an unexpired independent source in performing the method and the performance of the dry chemical is found to be satisfactory. The comparison must show that the dry chemical meets CCV limits. The comparison studies are maintained in the Standard Logbook in each laboratory group.

Wherever possible, standards must be traceable to national or international standards of measurement or to national or international reference materials. Records to that effect are available to the user.

Compressed gases in use are checked for pressure and secure positioning daily. The minimum total pressure must be 500 psig or the tank must be replaced. The quality of the gases must

meet method or manufacturer specification or be of a grade that does not cause any analytical interference.

Water used in the preparation of standards or reagents must have a conductivity of less than 1 mmho/cm (or specific resistivity of greater than 1.0 megaohm-cm) at 25^{o}C. The specific conductivity is checked and recorded daily. If the water's conductivity is greater than the specified limit, the Operations Manager and appropriate Department Managers/ Group Leaders must be notified immediately in order to notify all departments, decide on cessation (based on intended use) of activities, and make arrangements for correction.

The laboratory may purchase reagent grade (or other similar quality) water for use in the laboratory. This water must be certified "clean" by the supplier for all target analytes or otherwise verified by the laboratory prior to use. This verification is documented.

Standard lots are verified before first time use if the laboratory switches manufacturers or has historically had a problem with the type of standard.

Purchased VOA vials must be certified clean and the certificates must be maintained. If uncertified VOA vials are purchased, all lots must be verified clean prior to use. This verification must be maintained.

9.3.4 Storage

Reagent and chemical storage is important from the aspects of both integrity and safety. Light-sensitive reagents may be stored in brown-glass containers. Storage condictions are per the Corporate Environmental Health & Safety Manual (Corporate Document CW-E-M-001) and method SOPs or manufacturer instructions.

9.4 PURCHASE OF EQUIPMENT/INSTRUMENTS/SOFTWARE

When a new piece of equipment is needed, either for additional capacity or for replacing inoperable equipment, the analyst or group leader makes a supply request to the Operations Manager and/or the Laboratory Director/Manager. If they agree with the request the procedures outlined in TestAmerica's Corporate Policy CA-T-P-001, Qualified Products List, are followed. A decision is made as to which piece of equipment can best satisfy the requirements. The appropriate written requests are completed, and Purchasing places the order.

Upon receipt of a new or used piece of equipment, an identification name is assigned, such as HP-20, added to the equipment list described in Section 21 that is maintained by the QA Department, and I.T. must be notified so they can synchronize the instrument for backups. Its capability is assessed to determine if it is adequate or not for the specific application. For instruments, a calibration curve is generated followed by MDLs, Demonstration of Capabilities (DOCs), and other relevant criteria (refer to Section 20). For software, its operation must be deemed reliable and evidence of instrument verification must be retained by the IT Department or QA Department. Software certificates supplied by the vendors are filed with the LIMS Administrator. The manufacturer's operation manual is retained at the bench.

9.5 SERVICES

Service to analytical instruments (except analytical balances) is performed on an as needed basis. Routine preventative maintenance is discussed in Section 20. The need for service is determined by analysts and/or Department Managers. The service providers that perform the services are approved by the Department Managers or Operations Manager.

9.6 SUPPLIERS

TestAmerica selects vendors through a competitive proposal / bid process, strategic business alliances or negotiated vendor partnerships (contracts). This process is defined in the Corporate Finance documents on Vendor Selection (SOP CW-F-S-018) and Procurement and Contracts Policy (Policy CW-F-P-004). The level of control used in the selection process is dependent on the anticipated spending amount and the potential impact on TestAmerica business. Vendors that provide test and measuring equipment, solvents, standards, certified containers, instrument related service contracts or subcontract laboratory services shall be subject to more rigorous controls than vendors that provide off-the-shelf items of defined quality that meet the end use requirements. The JD Edwards purchasing system includes all suppliers /vendors that have been approved for use.

Evaluation of suppliers is accomplished by ensuring the supplier ships the product or material ordered and that the material is of the appropriate quality. This is documented by signing off on packing slips or other supply receipt documents. The purchasing documents contain the data that adequately describe the services and supplies ordered.

Any issues of vendor performance are to be reported immediately by the laboratory staff to the Corporate Purchasing Group by completing a Vendor Performance Report (CW-F-WI-009).

The Corporate Purchasing Group must work through the appropriate channels to gather the information required to clearly identify the problem and must contact the vendor to report the problem and to make any necessary arrangements for exchange, return authorization, credit, etc.

As deemed appropriate, the Vendor Performance Reports must be summarized and reviewed to determine corrective action necessary, or service improvements required by vendors

The laboratory has access to a listing of all approved suppliers of critical consumables, supplies and services. This information is provided through the JD Edwards purchasing system.

9.6.1 New Vendor Procedure

TestAmerica employees who wish to request the addition of a new vendor must complete a J.D. Edwards Vendor Add Request Form.

New vendors are evaluated based upon criteria appropriate to the products or services provided as well as their ability to provide those products and services at a competitive cost. Vendors are also evaluated to determine if there are ethical reasons or potential conflicts of interest with TestAmerica employees that would make it prohibitive to do business with them as well as their

financial stability. The QA Department and/or the Laboratory Director are consulted with vendor and product selection that have an impact on quality.

SECTION 10

COMPLAINTS
(NELAC 5.4.8)

10.1 OVERVIEW

The laboratory considers an effective client complaint handling process to be of significant business and strategic value. Listening to and documenting client concerns captures 'client knowledge' that enables our operations to continually improve processes and improving client satisfaction. An effective client complaint handling process also provides assurance to the data user that the laboratory will stand behind its data, service obligations and products.

A client complaint is any expression of dissatisfaction with any aspect of our business services, (e.g., communications, responsiveness, data, reports, invoicing and other functions) expressed by any party, whether received verbally or in written form. Client inquiries, complaints or noted discrepancies are documented, communicated to management, and addressed promptly and thoroughly.

The laboratory has procedures for addressing both external and internal complaints with the goal of providing satisfactory resolution to complaints in a timely and professional manner.

The nature of the complaint is identified, documented and investigated, and an appropriate action is determined and taken. In cases where a client complaint indicates that an established policy or procedure was not followed, the QA Department must evaluate whether a special audit must be conducted to assist in resolving the issue. A written confirmation or letter to the client, outlining the issue and response taken is recommended as part of the overall action taken.

The process of complaint resolution and documentation utilizes the procedures outlined in Section 12 (Corrective Actions) and is documented following SOPs NC-QA-029, Nonconformance and Corrective Action System, and CA-C-S-002, Complaint Handling and Service Recovery.

10.2 EXTERNAL COMPLAINTS

An employee that receives a complaint initiates the complaint resolution process by first documenting the complaint according to CA-C-S-002, Complaint Handling and Service Recovery.

Complaints fall into two categories: correctable and non-correctable. An example of a correctable complaint would be one where a report re-issue would resolve the complaint. An example of a non-correctable complaint would be one where a client complains that their data was repeatedly late. Non-correctable complaints must be reviewed for preventive action measures to reduce the likelihood of future occurrence and mitigation of client impact.

The general steps in the complaint handling process are:

- Receiving and Documenting Complaints
- Complaint Investigation and Service Recovery

- Process Improvement

The laboratory must inform the initiator of the complaint of the results of the investigation and the corrective action taken, if any.

10.3 INTERNAL COMPLAINTS

Internal complaints include, but are not limited to errors and non-conformances, training issues, internal audit findings, and deviations from methods. Corrective actions may be initiated by any staff member who observes a nonconformance and must follow the procedures outlined in Section 12. In addition, Corporate Management, Sales and Marketing, and Information Technology (IT) may initiate a complaint by contacting the laboratory or through the Corrective Action system described in Section 12.

10.4 MANAGEMENT REVIEW

The number and nature of client complaints is reported by the QA Manager to the laboratory and QA Director in the QA Monthly report. Monitoring and addressing the overall level and nature of client complaints and the effectiveness of the solutions is part of the Annual Management Review (Section 16)

SECTION 11

CONTROL OF NON-CONFORMING WORK
(NELAC 5.4.9)

11.1 OVERVIEW

When data discrepancies are discovered or deviations and departures from laboratory SOPs, policies, and/or client requests have occurred, corrective action is taken immediately. First, the laboratory evaluates the significance of the nonconforming work. Then, a Corrective Action plan is initiated based on the outcome of the evaluation. If it is determined that the nonconforming work is an isolated incident, the plan could be as simple as adding a qualifier to the final results and/or making a notation in the case narrative. If it is determined that the nonconforming work is a systematic or improper practices issue, the Corrective Action plan could include a more in depth investigation and a possible suspension of an analytical method. In all cases, the actions taken are documented using the laboratory's Corrective Action system (refer to Section 12).

Due to the frequently unique nature of environmental samples, sometimes departures from documented policies and procedures are needed.

Note: The laboratory must implement Corrective Action procedures to resolve the deviation and limit qualification of the final results. The laboratory is not permitted to deviate from its VAP approved SOP if it intends to attest under affidavit that the "results" are VAP certified. When all Corrective Actions listed in the SOP have been exhausted, it may be necessary to use technical judgment in which case the decision process and rationale will be presented in the final report and/or affidavit and the data will be noted as 'not VAP certified' on the affidavit**.**

11.2 RESPONSIBILITIES AND AUTHORITIES

TestAmerica's Corporate SOP entitled Internal Investigation of Potential Data Discrepancies and Determination for Data Recall (CA-L-S-001) outlines the general procedures for the reporting and investigation of data discrepancies and alleged incidents of misconduct or violations of the TestAmerica's data integrity policies as well as the policies and procedures related to the determination of the potential need to recall data.

Under certain circumstances the Laboratory Director/Manager, Operations Manager, Project Manager, or a member of the QA team may exceptionally authorize departures from documented procedures or policies. The departures may be a result of procedural changes due to the nature of the sample; a one-time procedure for a client; QC failures with insufficient sample to reanalyze, etc. In most cases, the client must be informed of the departure prior to the reporting of the data. Any departures must be well documented using the laboratory's Corrective Action procedures described in Section 13. This information may also need to be documented in logbooks and/or data review as appropriate*.* Any impacted data must be referenced in a case narrative and/or flagged with an appropriate data qualifier.

Any misrepresentation or possible misrepresentation of analytical data discovered by any laboratory staff member must be reported to facility Senior Management within 24 hours. The Senior Management staff is compromised of the Laboratory Director, QA Manager, Customer

Service Manager, Operations Manager, I.T. Manager, H.R. Manager, and Technical Director. The reporting of issues involving alleged violations of the company's Data Integrity or Manual Integration procedures must be conveyed to an Ethics and Compliance Officer (ECO), Director of Quality and Client Advocacy, and the laboratory's Corporate Quality Director within 24 hours of discovery.

Whether an inaccurate result was reported due to calculation or quantitation errors, data entry errors, improper practices, or failure to follow SOPs, the data must be evaluated to determine the possible effect.

The Laboratory Director/Manager, QA Manager, ECOs, Corporate Quality Director, the COO, General Managers, and the Corporate Quality Directors have the authority and responsibility to halt work, withhold final reports, or suspend an analysis for due cause as well as authorize the resumption of work.

11.3 EVALUATION OF SIGNIFICANCE AND ACTIONS TAKEN

For each nonconforming issue reported, an evaluation of its significance and the level of management involvement needed is made. This includes reviewing its impact on the final data, whether or not it is an isolated or systematic issue, and how it relates to any special client requirements.

TestAmerica's Corporate Data Investigation and Recall Procedure (SOP CA-L-S-001) distinguishes between situations when it would be appropriate for laboratory management to make the decision on the need for client notification (written or verbal) and data recall (report revision) and when the decision must be made with the assistance of the ECOs and Corporate Management. Laboratory level decisions are documented and approved using the laboratory's standard nonconformance/Corrective Action reporting in lieu of the data recall determination form contained in TestAmerica Corporate SOP CA-L-S-001.

11.4 PREVENTION OF NONCONFORMING WORK

If it is determined that the nonconforming work could recur, further corrective actions must be made following the laboratory's Corrective Action system. On a monthly basis, the QA Department evaluates non-conformances to determine if any nonconforming work has been repeated multiple times. If so, the laboratory's Corrective Action process may be followed.

11.5 METHOD SUSPENSION/RESTRICTION (STOP WORK PROCEDURES)

In some cases it may be necessary to suspend/restrict the use of a method or target compound which constitutes significant risk and/or liability to the laboratory. Suspension/restriction procedures can be initiated by any of the persons noted in Section 11.2, Paragraph 5.

Prior to suspension/restriction, confidentiality must be respected, and the problem with the required corrective and preventive action must be stated in writing and presented to the Laboratory Director/Manager.

The Laboratory Director/Manager must arrange for the appropriate personnel to meet with the QA Manager as needed. This meeting must be held to confirm that there is a problem, that suspension/restriction of the method is required and must be concluded with a discussion of the steps necessary to bring the method/target or test fully back on line. In some cases that may not be necessary if all appropriate personnel have already agreed there is a problem and there is agreement on the steps needed to bring the method, target, or test fully back on line.

The QA Manager must also initiate a Corrective Action report as described in Section 12 if one has not already been started. A copy of any meeting notes and agreed-upon steps should be faxed or e-mailed by the laboratory to the appropriate General Manager and member of Corporate QA. This fax/e-mail acts as notification of the incident.

After suspension/restriction, the lab will hold all reports to clients pending review. No faxing, mailing or distributing through electronic means may occur. The report must not be posted for viewing on the Internet. It is the responsibility of the Laboratory Director/Manager to hold all reporting and to notify all relevant laboratory personnel regarding the suspension/restriction, i.e., Project Management, Log-in, etc. Clients must NOT generally be notified at this time. Analysis may proceed in some instances depending on the non-conformance issue.

Within 72 hours, the QA Manager must determine if compliance is now met and reports can be released, OR determine the plan of action to bring work into compliance, and release work. A team, with all principals involved (Laboratory Director, Technical Director, QA Manager, Group Leader) can devise a start-up plan to cover all steps from client notification through compliance and release of reports. Project Management, and the Directors of Client Services and Sales and Marketing must be notified if clients must be notified or if the suspension/restriction affects the laboratory's ability to accept work. The QA Manager must approve start-up or elimination of any restrictions after all corrective action is complete. This approval is given by final signature on the completed Corrective Action report.

SECTION 12

CORRECTIVE ACTION
(NELAC 5.4.10)

12.1 OVERVIEW

A major component of TestAmerica's Quality Assurance (QA) Program is the problem investigation and feedback mechanism designed to keep the laboratory staff informed on quality related issues and to provide insight to problem resolution. When nonconforming work or departures from policies and procedures in the quality system or technical operations are identified, the Corrective Action procedure provides a systematic approach to assess the issues, restore the laboratory's system integrity, and prevent reoccurrence. Corrective actions are documented using Nonconformance Memos (NCM).

12.2 GENERAL

Problems within the quality system or within analytical operations may be discovered in a variety of ways, such as QC sample failures, internal or external audits, proficiency testing (PT) performance, client complaints, staff observation, etc.

The purpose of a Corrective Action system is to:

- Identify non-conformance events and assign responsibility(s) for investigating.
- Resolve non-conformance events and assign responsibility for any required corrective action.
- Identify systematic problems before they become serious.
- Identify and track client complaints and provide resolution (see more on client complaints in Section 11).

12.2.1 Non-Conformance Memo (NCM)

An NCM is used to document the following types of corrective actions:

- Deviations from an established procedure or SOP
- QC outside of limits (non matrix related)
- Isolated Reporting / Calculation Errors
- Client Complaints
- Discrepancies in materials / goods received vs. manufacturer packing slips

12.3 CLOSED LOOP CORRECTIVE ACTION PROCESS

Any employee in the company can initiate a Corrective Action. There are four main components to a closed-loop Corrective Action process once an issue has been identified--Cause Analysis, Selection and Implementation of Corrective Actions (both short and long term), Monitoring of the Corrective Actions, and Follow-up.

12.3.1 Cause Analysis

Upon discovery of a non-conformance event, the event must be defined and documented. An NCM must be initiated, someone is assigned to investigate the issue, and the event is investigated for cause. Table 12-1 provides some general guidelines on determining responsibility for assessment. SOP NC-QA-029, Nonconformance and Corrective Action System, establishes procedures for the identification and documentation of nonconformances and the corrective actions taken as a result of these events.

The cause analysis step is the key to the process as a long-term corrective action cannot be determined until the cause is determined.

If the cause is not readily obvious, the Group Leader, Lab Director, QA Manager, or designee is consulted.

12.3.2 Selection and Implementation of Corrective Actions

Where corrective action is needed, the laboratory must identify potential corrective actions. The action(s) most likely to eliminate the problem and prevent recurrence are selected and implemented. Responsibility for implementation is assigned.

Corrective actions must be, to a degree, appropriate to the magnitude of the problem identified through the cause analysis.

Whatever corrective action is determined to be appropriate, the laboratory must document and implement the changes. The NCM is used for this documentation.

12.3.3 Root Cause Analysis

Root Cause Analysis is a class of problem solving (investigative) methods aimed at identifying the basic or causal factor(s) that underlie variation in performance or the occurrence of a significant failure. The root cause may be buried under seemingly innocuous events, many steps preceding the perceived failure. At first glance, the immediate response is typically directed at a symptom and not the cause. Typically, root cause analysis would be best with three or more incidents to triangulate a weakness.

Systematically analyze and document the Root Causes of the more significant problems that are reported. Identify, track, and implement the corrective actions required to reduce the likelihood of recurrence of significant incidents. Trend the Root Cause data from these incidents to identify root causes that, when corrected, can lead to dramatic improvements in performance by eliminating entire classes of problems.

Identify the one event associated with problem and ask why this event occurred. Brainstorm the root causes of failures by asking why events occurred or conditions existed; and then why the cause occurred 5 consecutive times until you get to the root cause. For each of these sub events or causes, ask why it occurred. Repeat the process for the other events associated with the incident.

- Root cause analysis does not mean the investigation is over. Look at technique, or other systems outside the normal indicators. Often creative thinking will find root causes that ordinarily would be missed, and continue to plague the laboratory or operation.

12.3.4 Monitoring of the Corrective Actions

- The Department Manager/Group Leader and QA Manager is responsible to ensure the corrective action taken was effective.
- Ineffective actions are documented and re-evaluated until acceptable resolution is achieved. Department Managers are accountable to the Laboratory Director to ensure final acceptable resolution is achieved and documented appropriately.
- Each NCM is entered into a database for tracking purposes and a monthly summary of all corrective actions is printed out for review to aid in ensuring the corrective actions have taken effect.
- The QA Manager reviews monthly NCMs for trends. Highlights are included in the QA monthly report (refer to Section 16). If a significant trend develops that adversely affects quality, an audit of the area is performed and corrective action implemented.
- Any out-of-control situations that are not addressed acceptably at the laboratory level may be reported to the Corporate Quality Director by the QA Manager, indicating the nature of the out-of-control situation and problems encountered in solving the situation.

12.3.5 Follow-up Audits

- Follow-up audits may be initiated by the QA Manager and must be performed as soon as possible when the identification of a nonconformance casts doubt on the laboratory's compliance with its own policies and procedures, or on its compliance with state or federal requirements.
- These audits often follow the implementation of the corrective actions to verify effectiveness. An additional audit would only be necessary when a critical issue or risk to business is discovered.

Also refer to Section 15.2.4, Special Audits.)

12.4 TECHNICAL CORRECTIVE ACTIONS

In addition to providing acceptance criteria and specific protocols for technical corrective actions in the method SOPs, the laboratory has general procedures to be followed to determine when departures from the documented policies and procedures and quality control have occurred (refer to Section 11 for information regarding the control of non-conforming work). The documentation of these procedures is through the use of an NCM.

Table 12-1 includes examples of general technical corrective actions. For specific criteria and corrective actions, refer to the analytical methods or specific method SOPs.

Table 12-1 provides some general guidelines for identifying the individual(s) responsible for assessing each QC type and initiating corrective action. The table also provides general guidance on how a data set should be treated if associated QC measurements are unacceptable. Specific procedures are included in Method SOPs, Work Instructions, and QAM Sections 19 and 20. The QA Manager reviews all corrective actions monthly, at a minimum, and highlights are included in the QA monthly report.

To the extent possible, samples must be reported only if all quality control measures are acceptable. If the deficiency does not impair the usability of the results, data must be reported with an appropriate data qualifier and/or the deficiency must be noted in the case narrative. Where sample results may be impaired, the Project Manager is notified by a written NCM and appropriate corrective action (e.g., re-analysis) is taken and documented**.**

12.5 BASIC CORRECTIONS

When mistakes occur in records, each mistake must be crossed-out, and [not obliterated (e.g. no White-Out)], and the correct value entered alongside. All such corrections must be initialed (or signed) and dated by the person making the correction. In the case of records stored electronically, the original “uncorrected” file must be maintained intact and a second “corrected” file is created.

This same process applies to adding additional information to a record. All additions made later than the initial must also be initialed (or signed) and dated.

When corrections are due to reasons other than obvious transcription errors, the reason for the corrections (or additions) must also be documented.

Table 12-1 .
General Corrective Action Procedures

*** For the Ohio EPA Voluntary Action Program (VAP), please refer to the SOPs for the acceptable criteria, corrective actions, and exceptions.**

INORGANIC LABORATORY QUALITY CONTROL SAMPLES

Analysis	QC Sample	Method	NPDES [1]	Method	RCRA (SW846) [2]
Alkalinity	* Method Blank	310.1 2320B	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration must be less than the reporting limit Corrective Action: Rerun all samples associated with unacceptable blank	—	N/A
	Laboratory Control Sample	310.1 2320B	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: If not within laboratory control limits, rerun all associated samples	—	N/A
	Matrix Spike	310.1 2320B	Total alkalinity: 1 per batch of 20 samples	—	N/A
	Matrix Spike Duplicate	310.1 2320B	Total alkalinity: 1 per batch of 20 samples	—	N/A
	Duplicate	310.1 2320B	For carbonate, bicarbonate, hydroxide, alkalinity only. Frequency: 1 per batch of 10 samples Criteria 310.1: ≤ 20 % RPD(3) Criteria 2320B: ≤ 25 % RPD(3) Corrective Action: Flag data outside of limit.	—	N/A

INORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES [1]	Method	RCRA (SW846) [2]
Ammonia	* Method Blank	350.1 350.2 SM4500 NH-E,F	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration less than reporting limit Corrective Action: Rerun all samples associated with unacceptable blank	—	N/A
	Laboratory Control Sample	350.1 350.2 SM4500 NH-E,F	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: If not within control limits, rerun all associated samples	—	N/A
	Matrix Spike	350.1 350.2 SM4500 NH-E,F	Frequency: 1 per 20 samples, minimum of one per batch of samples processed Criteria: Percent recovery must be within laboratory control limits Corrective Action: Flag data outside of limit	—	N/A

INORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES [1]	Method	RCRA (SW846) [2]
Ammonia (Cont'd)	Matrix Spike Duplicate	350.1 350.2 SM4500 NH-E,F	Frequency: 1 per 20 samples, minimum of one per batch of samples processed Criteria: Percent recovery must be within laboratory control limits Corrective Action: Flag data outside of limit	—	N/A
	Duplicate	350.1 350.2 SM4500 NH-E,F	N/A	—	N/A
Ammonia (TKN)	* Method Blank	351.2 351.3 SM4500 NO_3	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration must be less than the reporting limit Corrective Action: Rerun all samples associated with unacceptable blank	—	N/A

INORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES [1]	Method	RCRA (SW846) [2]
Ammonia (TKN) (Cont'd)	Laboratory Control Sample	351.2 351.3 SM4500 NO_3	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: If not within laboratory control limits, rerun all associated samples	—	N/A
	Matrix Spike	351.2 351.3 SM4500 NO_3	Frequency: 1 per 20 samples, minimum of one per batch of samples processed Criteria: Must be within laboratory control limits Corrective Action: Flag data outside of limit	—	N/A
	Matrix Spike Duplicate	351.2 351.3 SM4500 NO_3	Frequency: 1 per 20 samples, minimum of one per batch of samples processed Criteria: Must be within laboratory control limits Corrective Action: Flag data outside of limit	—	N/A
	Duplicate	351.2 351.3 SM4500 NO_3	N/A	—	N/A

INORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES [1]	Method	RCRA (SW846) [2]
BOD	* Method Blank	405.1 SM5210B	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration must be less than the reporting limit Corrective Action: Rerun all samples associated with unacceptable blank	—	N/A
	Laboratory Control Sample and Laboratory Control Sample Duplicate	405.1 SM5210B	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: If not within laboratory control limits, rerun all associated samples	—	N/A
	Matrix Spike	405.1 SM5210B	N/A	—	N/A
	Matrix Spike Duplicate	405.1 SM5210B	N/A	—	N/A
	Duplicate	405.1 SM5210B	N/A	—	N/A

INORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES [1]	Method	RCRA (SW846) [2]
Bromide	* Method Blank	300.0A[(5)]	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration must be less than the reporting limit Corrective Action: Rerun all samples associated with unacceptable blank	9056A	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration must be less than the reporting limit Corrective Action: Rerun all samples associated with unacceptable blank
	Laboratory Control Sample and Laboratory Control Sample Duplicate	300.0A[(5)]	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: If not within control limits, rerun all associated samples	9056A	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: If not within control limits, rerun all associated samples
	Matrix Spike	300.0A[(5)]	Frequency: 1 per 10 samples, minimum of one per batch of samples processed Criteria: Percent recovery must be within laboratory control limits Corrective Action: Flag data outside of limit	9056A	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: Flag data associated with MS outside of limit
	Matrix Spike Duplicate	300.0A[(5)]	N/A	9056A	N/A
	Duplicate	300.0A[(5)]	N/A	9056A	N/A

INORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES [1]	Method	RCRA (SW846) [2]
Chemical Oxygen Demand (COD)	* Method Blank	410.4 SM5220D	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration must be less than the reporting limit Corrective Action: Rerun all samples associated with unacceptable blank	—	N/A
	Laboratory Control Sample and Laboratory Control Sample Duplicate	410.4 SM5220D	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: If not within laboratory control limits, rerun all associated samples	—	N/A
	Matrix Spike	410.4 SM5220D	Frequency: 1 per 10 samples, minimum of one per batch of samples processed Criteria: Must be within laboratory control limits Corrective Action: Flag data outside of limit	—	N/A
	Matrix Spike Duplicate	410.4 SM5220D	Frequency: 1 per 20 samples, minimum of one per batch of samples processed Criteria: Must be within laboratory control limits Corrective Action: Flag data outside of limit	—	N/A
	Duplicate	410.4 SM5220D	N/A	—	N/A

INORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES [1]	Method	RCRA (SW846) [2]
Chloride	* Method Blank	300.0A [5] 325.2 325.3	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration must be less than the reporting limit Corrective Action: Rerun all samples associated with unacceptable blank	9056A	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration must be less than the reporting limit Corrective Action: Rerun all samples associated with unacceptable blank
	Laboratory Control Sample	300.0A [5] 325.2 325.3	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: If not within control limits, rerun all associated samples	9056A	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: If not within laboratory control limits, rerun all associated samples
	Matrix Spike	300.0A(5)	Frequency: 1 per 10 samples, minimum of one per batch of samples processed Criteria: Percent recovery must be within laboratory control limits Corrective Action: Flag data outside of limit	9056A	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Methods 9251Corrective Action: If not within laboratory control limits, rerun all associated samples Method 9056/9253 Corrective Action: Flag data associated with MS outside of limits

INORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES [1]	Method	RCRA (SW846) [2]
Chloride (cont'd)	Matrix Spike Duplicate	325.2 325.3	Frequency: 1 per 20 samples, minimum of one per batch of samples processed Criteria: Percent recovery must be within laboratory control limits Corrective Action: Flag data outside of limit	9056A	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits/< 20 % RPD(3) Corrective Action: Flag data outside of limit Method 9056: MSD is not applicable
	Duplicate	300.0A(5) 325.2 325.3	N/A	9056A	N/A
Chlorine, Residual	* Method Blank	330.5	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration must be less than the reporting limit Corrective Action: Rerun all samples associated with unacceptable blank	—	N/A
	Laboratory Control Sample	330.5	N/A	—	N/A
	Matrix Spike	330.5	N/A	—	N/A
	Matrix Spike Duplicate	330.5	N/A	—	N/A
	Duplicate	330.5	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: ≤ 20 % RPD(3) Corrective Action: Flag data outside of limit.	—	Water

INORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES [1]	Method	RCRA (SW846) [2]
Chromium (Cr^{+6})	* Method Blank	3500 Cr-D	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration must be less than the reporting limit Corrective Action: Rerun all samples associated with unacceptable blank unless the blank is above RL, and samples are ND.	7196A 3060A	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration less than reporting limit Corrective Action: Rerun all samples associated with unacceptable blank unless the blank is above RL, and samples are ND.
	Laboratory Control Sample	3500 Cr-D	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: If not within laboratory control limits, rerun all associated samples	7196A 3060A	Frequency: 1 with each batch of samples processed not to exceed 20 samples prepped Criteria: percent recovery for water must be within ± 15 % and for solids must be within ± 20% Corrective Action: Rerun all samples associated with unacceptable LCS
	Matrix Spike	3500 Cr-D	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Must be within laboratory QC limits Corrective Action: Flag data outside of limit	3060A 7196A	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Advisory limits are 75% - 125% recovery Corrective Action: Flag data associated with unacceptable Matrix Spike

INORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES [1]	Method	RCRA (SW846) [2]
Chromium (Cr^{+6}) (Cont'd)	Matrix Spike Duplicate	3500 Cr-D	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Must be within laboratory QC limits Corrective Action: Flag data outside of limit	7196A 3060A	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Advisory limits are 75% - 125% recovery Corrective Action: Flag data associated with unacceptable Matrix Spike
	Duplicate	3500 Cr-D	N/A	7196A 3060A	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: ≤ 20 % RPD(3) limit Corrective Action: Flag data outside of limit.

INORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES [1]	Method	RCRA (SW846) [2]
Conductivity, Specific	* Method Blank	120.1 SM2510B	N/A	9050A	Not Applicable
	Laboratory Control Sample	120.1 SM2510B	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: If not within laboratory control limits, rerun all associated samples	9050A	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: If not within laboratory control limits, rerun all associated samples
	Matrix Spike	120.1 SM2510B	N/A	9050A	N/A
	Matrix Spike Duplicate	120.1 SM2510B	N/A	9050A	N/A
	Duplicate	120.1 SM2510B	Frequency: 1 with each batch of samples processed not to exceed 10 samples Criteria: ≤ 20 % RPD(3) Corrective Action: Flag data outside of limit.	9050A	Frequency: 1 with each batch of samples processed not to exceed 10 samples

INORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES [1]	Method	RCRA (SW846) [2]
Cyanide (Amenable)	* Method Blank	335.1 SM4500C N-G	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration must be less than the reporting limit Corrective Action: Rerun all samples associated with unacceptable blank	9012A	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration less than reporting limit Corrective Action: Rerun all samples associated with unacceptable blank
	Laboratory Control Sample	335.1 SM4500C N-G	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: If not within laboratory control limits, rerun all associated samples	9012A	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: Rerun all samples associated with unacceptable LCS
	Matrix Spike	335.1 SM4500C N-G	N/A	9012A	N/A
	Matrix Spike Duplicate	335.1 SM4500C N-G	N/A	9012A	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within lab control limits Corrective Action: Flag data associated with unacceptable Matrix Spike
	Duplicate	335.1 SM4500C N-G	N/A	9012A	N/A

INORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES [1]	Method	RCRA (SW846) [2]
Cyanide (Total)	* Method Blank	335.2 335.3 335.4 4500-CN E 335.2-CLP-M	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration must be less than the reporting limit Corrective Action: Rerun all samples associated with unacceptable blank unless the blank is above RL, and samples are ND.	9012A	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration less than reporting limit Corrective Action: Rerun all samples associated with unacceptable blank unless the blank is above RL, and samples are ND.
	Laboratory Control Sample	335.2 335.3 335.4 4500-CN E 335.2-CLP-M	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: If not within laboratory control limits, rerun all associated samples	9012A	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: Rerun all samples associated with unacceptable LCS
	Matrix Spike	335.2 335.3 335.4 4500-CN E 335.2-CLP-M	Frequency: 1 per 20 samples, minimum of one per batch of samples processed Criteria: Percent recovery must be within laboratory control limits Corrective Action: Flag data outside of limit	9012A	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: Flag data associated with unacceptable Matrix Spike

INORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES [1]	Method	RCRA (SW846) [2]
Cyanide (Total) (cont'd)	Matrix Spike Duplicate	335.2 335.3 335.4 4500-CN E 335.2-CLP-M	Frequency: 1 per 20 samples, minimum of one per batch of samples processed Criteria: Percent recovery must be within laboratory control limits Corrective Action: Flag data outside of limit	9012A	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: Flag data associated with unacceptable Matrix Spike
	Duplicate	335.2 335.3 335.4 335.2-CLP-M	N/A	9012A	N/A
Flashpoint	* Method Blank	—	N/A	1010 ASTM D93-9	N/A
	Laboratory Control Sample	—	N/A	1010 ASTM D93-9	N/A
	Matrix Spike	—	N/A	1010 ASTM D93-9	N/A
	Matrix Spike Duplicate	—	N/A	1010 ASTM D93-9	N/A
	Duplicate	—	Frequency: 1 per batch of ≤20 samples per matrix Criteria: RPD(3) must be ≤ 20% Corrective Action: Flag data associated with unacceptable Duplicate	1010 ASTM D93-9	Frequency: 1 per batch of ≤20 samples per matrix Criteria: RPD(3) must be ≤ 20% Corrective Action: Flag data associated with unacceptable Duplicate

INORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES [1]	Method	RCRA (SW846) [2]
Fluoride	* Method Blank	300.0A[(5)] 340.2 SM4500F-C,ISE	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration must be less than the reporting limit Corrective Action: Rerun all samples associated with unacceptable blank	9056A	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration must be less than the reporting limit Corrective Action: Rerun all samples associated with unacceptable
	Laboratory Control Sample	300.0A[(5)] 340.2 SM4500F-C,ISE	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: If not within laboratory control limits, rerun all associated samples	9056A	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: If not within control limits, rerun all associated samples
	Laboratory Control Sample Duplicate	300.0A[(5)] SM4500F-C,ISE	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: If not within laboratory control limits, rerun all associated samples	9056	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: If not within control limits, rerun all associated samples

INORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES [1]	Method	RCRA (SW846) [2]
Flouride (Cont'd)	Matrix Spike	300.0A[(5)] 340.2 SM4500F-C,ISE	Frequency: 1 per 10 samples by IC Criteria: Must be within laboratory QC limits Corrective Action: Flag data outside of limit	9056A	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: Flag data associated with outside of limit
	Matrix Spike Duplicate	340.2 SM4500F-C,ISE	Frequency: 1 per 20 samples by IC Criteria: Must be within laboratory QC limits Corrective Action: Flag data outside of limit	340.2	N/A
	Duplicate	300.0A[(5)] 340.2 SM4500F-C,ISE	N/A	9056A	Frequency: 1 with each batch of samples processed Criteria: RPD[(3)] must be within laboratory control limits Corrective Action: Flag data associated with duplicates outside of laboratory RPD[(3)] limits

INORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES [1]	Method	RCRA (SW846) [2]
Hardness	* Method Blank	130.2 2340B	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration must be less than the reporting limit Corrective Action: Rerun all samples associated with unacceptable blank	—	N/A
	Laboratory Control Sample	130.2 2340B	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: If not within laboratory control limits, rerun all associated samples	—	N/A
	Matrix Spike	130.2 2340B	Method 130.2: 1 per 20 samples Method 2340B: Frequency , Criteria, and Corrective Action: See ICP Metals Method 200.7 Requirements	—	N/A

INORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES [1]	Method	RCRA (SW846) [2]
Hardness (cont'd)	Matrix Spike Duplicate	130.2 2340B	Method 130.2: 1 per 20 samples Method 2340B: Frequency, Criteria, and Corrective Action: See ICP Metals Method 200.7 Requirements	—	N/A
	Duplicate	130.2 2340B	N/A	—	N/A
Iron, Ferrous & Ferric	* Method Blank	3500-Fe D	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration must be less than the reporting limit Corrective Action: Rerun all samples associated with unacceptable blank	—	N/A
	Laboratory Control Sample	3500-Fe D	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: If not within laboratory control limits, rerun all associated samples	—	N/A

INORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES [1]	Method	RCRA (SW846) [2]
Iron, Ferrous & Ferric (Cont'd)	Matrix Spike	3500-Fe D	Frequency: 1 every 10 samples Criteria: Must be within laboratory QC limits Corrective Action: Flag associated data outside of limit	—	N/A
	Matrix Spike Duplicate	3500-Fe D	Frequency: 1 every 10 samples Criteria: Must be within laboratory QC limits Corrective Action: Flag associated data outside of limit	—	N/A
	Duplicate	3500-Fe D	N/A	—	N/A
Nitrate	* Method Blank	300.0A(5) 353.2 SM4500-NO_3-E	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration must be less than the reporting limit Corrective Action: Rerun all samples associated with unacceptable blank	9056A	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration must be less than the reporting limit

INORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES [1]	Method	RCRA (SW846) [2]
Nitrate (Cont'd)	Laboratory Control Sample	300.0A[(5)] 353.2 SM4500-NO_3-E	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: If not within laboratory control limits, rerun all associated samples	9056A	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: If not within laboratory control limits, rerun all associated samples
	Matrix Spike	300.0A[(5)] 353.2 SM4500-NO_3-E	Frequency: 1 per 10 samples, minimum of one per batch of samples processed Criteria: Must be within laboratory control limits Corrective Action: Flag data outside of limit	9056A	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: If not within laboratory control limits, flag all associated samples
	Matrix Spike Duplicate	300.0A[(5)] 353.2 SM4500-NO_3-E	Frequency: 1 per 20 samples, minimum of one per batch of samples processed Criteria: Must be within laboratory control limits Corrective Action: Flag data outside of limit	9056A	N/A

INORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES [1]	Method	RCRA (SW846) [2]
Nitrate (cont'd)	Duplicate	300.0A(5) 353.2 SM4500-NO_3-E	N/A	9056A	Frequency: 1 per 10 samples Criteria: RPD(3) must be within laboratory control limits Corrective Action: If not within laboratory control limits, flag all associated samples
Nitrite	* Method Blank	300.0A(5) 353.2	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration must be less than the reporting limit Corrective Action: Rerun all samples associated with unacceptable blank	9056A	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration must be less than the reporting limit Corrective Action: Rerun all samples associated with unacceptable blank
	Laboratory Control Sample	300.0A(5) 354.1 353.2	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: If not within laboratory control limits, rerun all associated samples	9056A	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: If not within laboratory control limits, rerun all associated samples

INORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES [1]	Method	RCRA (SW846) [2]
Nitrite (Cont'd)	Matrix Spike	300.0A[(5)] 354.1 353.2	Frequency: 1 per 10 samples, minimum of one per batch of samples processed Criteria: Must be within laboratory control limits Corrective Action: Flag data outside of limit	9056A	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: If not within laboratory control limits, flag all associated samples
	Matrix Spike Duplicate	300.0A[(5)] 354.1 353.2	Frequency: 1 per 20 samples, minimum of one per batch of samples processed Criteria: Must be within laboratory control limits Corrective Action: Flag data outside of limit	9056A	N/A
	Duplicate	300.0A[(5)] 354.1 353.2	N/A	9056A	Frequency: 1 per 10 samples Criteria: RPD[(3)] must be within laboratory control limits Corrective Action: If not within laboratory control limits, flag all associated samples

INORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES [1]	Method	RCRA (SW846) [2]
Nitrate-Nitrite	* Method Blank	353.2	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration must be less than the reporting limit Corrective Action: Rerun all samples associated with unacceptable blank	—	N/A
	Laboratory Control Sample	353.2	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: If not within laboratory control limits, rerun all associated samples	—	N/A
	Matrix Spike	353.2	Frequency: 1 per 20 samples, minimum of one per batch of samples processed Criteria: Percent recovery must be within laboratory control limits Corrective Action: Flag data outside of limit	—	N/A
	Matrix Spike Duplicate	353.2	Frequency: 1 per 20 samples, minimum of one per batch of samples processed Criteria: Percent recovery must be within laboratory control limits Corrective Action: Flag data outside of limit	—	N/A
	Duplicate	353.2	N/A	—	N/A

INORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES [1]	Method	RCRA (SW846) [2]
pH	* Method Blank	150.1 SM4500H-B	N/A	9040B 9045C	N/A
	Laboratory Control Sample	150.1 SM4500H-B	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: If not within laboratory control limits, rerun all associated samples	9040B 9045C	N/A
	Matrix Spike	150.1 SM4500H-B	N/A	9040B 9045C	N/A
	Matrix Spike Duplicate	150.1 SM4500H-B	N/A	9040B 9045C	N/A
	Duplicate	150.1 SM4500H-B	Frequency: 1 with each batch of samples processed not to exceed 10 samples per matrix Criteria: ≤ 20 % RPD(3) limit Corrective Action: Flag data outside of limit.	9040B 9045C	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Advisory limits are ≤ 20% RPD(3) Corrective Action: Flag data associated with unacceptable Duplicate
Phenolics	* Method Blank	420.1	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration must be less than the reporting limit Corrective Action: Rerun all samples associated with unacceptable blank	9065	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration less than reporting limit Corrective Action: Rerun all samples associated with unacceptable blank

INORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES [1]	Method	RCRA (SW846) [2]
Phenolics (Cont'd)	Laboratory Control Sample	420.1	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: If not within laboratory control limits, rerun all associated samples	9065	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: If not within laboratory control limits, rerun all associated samples
	Matrix Spike	420.1	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: Flag data associated with unacceptable matrix spike	9065	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: Flag associated data
	Matrix Spike Duplicate	420.1	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: Flag associated data	9065	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: Flag associated data

INORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES [1]	Method	RCRA (SW846) [2]
Phosphorus (Total and Ortho-phosphate)	* Method Blank	300.0 (4,5) 365.3 365.1 SM4500P-E	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration must be less than the reporting limit Corrective Action: Rerun all samples associated with unacceptable blank	9056A	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration less than reporting limit Corrective Action: Rerun all samples associated with unacceptable blank
	Laboratory Control Sample	300.0(4,5) 365.3 365.1 SM4500P-E	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: If not within laboratory control limits, rerun all associated samples	9056A	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: If not within laboratory control limits, rerun all associated samples
	Matrix Spike	300.0(4,5) 365.3 365.1 SM4500P-E	Frequency: 1 per 10 samples Criteria: Must be within laboratory QC limits Corrective Action: Flag data outside of limit	9056A	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: Flag associated data associated with MS outside of limits
	Matrix Spike Duplicate	365.3 365.1 SM4500P-E	Frequency: 1 per 20 samples Criteria: Must be within laboratory QC limits Corrective Action: Flag data outside of limit	9056A	**N/A**

INORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES [1]	Method	RCRA (SW846) [2]
Phosphorus (Total and Ortho-phosphate) (Cont'd)	Duplicate	300.0(4,5) 365.3 365.1 SM4500P-E	N/A	9056A	Frequency: 1 with each batch of samples processed Criteria: RPD(3) must be within laboratory control limits Corrective Action: Flag data associated with duplicates outside of laboratory RPD(3) limits
Solids	* Method Blank	160.1 160.2 160.3 160.4 160.5 SM2540E	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration must be less than the reporting limit Corrective Action: If analyte level in method blank is ≥ RL for the analyte of interest in the sample, all associated samples with reportable levels of analyte are reprepared and reanalyzed.	—	N/A
	Laboratory Control Sample	160.1 160.2 160.3 160.4 160.5 SM2540E	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: If not within laboratory control limits, reprepare and rerun all associated samples	—	N/A

INORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES [1]	Method	RCRA (SW846) [2]
Solids (Cont'd)	Matrix Spike	160.1 160.2 160.3 160.4 160.5 SM4500P-E	N/A	—	N/A
	Matrix Spike Duplicate	160.1 160.2 160.3 160.4 160.5 SM4500P-E	N/A	—	N/A
	Duplicate	160.1 160.2 160.3 160.4 160.5 SM4500P-E	Frequency: 1 with each batch of samples processed not to exceed 10 samples Criteria: Sample results should agree within 20% if both the sample and sample duplicate results are > 5 X RL Corrective Action: Flag data outside of limit	—	N/A

INORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES [1]	Method	RCRA (SW846) [2]
Sulfate	* Method Blank	300.0A[(5)] 375.4	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration must be less than the reporting limit Corrective Action: Rerun all samples associated with unacceptable blank	9038 9056A	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration must be less than the reporting limit Corrective Action: Rerun all samples associated with unacceptable blank
	Laboratory Control Sample	300.0A[(5)] 375.4	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: If not within laboratory control limits, rerun all associated samples	9038 9056A	Frequency: 1 with each batch of samples processed not to exceed 20 samples Method 9038 Criteria: Percent recovery must be within ± 15 % Method 9056 Criteria: Percent recovery must be within laboratory control limits Corrective Action: Rerun all samples associated with unacceptable LCS (ICV)

INORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES [1]	Method	RCRA (SW846) [2]
Sulfate (cont'd)	Matrix Spike	300.0A[(5)] 375.4	Frequency: 1 per 10 samples, minimum of one per batch of samples processed Criteria: Percent recovery must be within laboratory control limits Corrective Action: Flag data outside of limit	9038 9056A	Frequency: 1 with each batch of samples processed not to exceed 10 samples (9038) or 20 samples (9056) Method 9038 Criteria: Limits are 75% - 125% recovery Method 9056 Criteria: Percent recovery must be within laboratory 0control limits Corrective Action: Flag data associated with unacceptable Matrix Spike
	Matrix Spike Duplicate	300.0A[(5)] 375.4	Frequency: 1 per 20 samples, minimum of one per batch of samples processed Criteria: Percent recovery must be within laboratory control limits Corrective Action: Flag data outside of limit	9038 9056A	N/A
	Duplicate	300.0A[(5)] 375.4	N/A	9038 9056A	N/A
Sulfide	* Method Blank	376.1	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration must be less than the reporting limit Corrective Action: Rerun all samples associated with unacceptable blank	9030A	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration must be less than the reporting limit Corrective Action: Rerun all samples associated with unacceptable blank

INORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES [1]	Method	RCRA (SW846) [2]
Sulfide (Cont'd)	Laboratory Control Sample	376.1	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: If not within laboratory control limits, rerun all associated samples	9030A	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: Flag associated data
	Matrix Spike	376.1	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: Flag data outside of limit	9030A	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: Flag associated data
	Matrix Spike Duplicate	376.1	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: Flag data outside of limit	9030A	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: Flag associated data Method 9034: Not Applicable
	Duplicate	376.1	N/A	9030A	N/A

INORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES [1]	Method	RCRA (SW846) [2]
Total Organic Carbon (TOC)	* Method Blank	415.1 SM5310D	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration must be less than the reporting limit Corrective Action: Rerun all samples associated with unacceptable blank	9060 Walkley-Black	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration less than reporting limit Corrective Action: Rerun all samples associated with unacceptable blank
	Laboratory Control Sample	415.1 SM5310D	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: If not within laboratory control limits, rerun all associated samples	9060 Walkley-Black	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: percent recovery must be within laboratory control limits Corrective Action: Rerun all samples associated with unacceptable LCS
	Matrix Spike	415.1 SM5310D	Frequency: 1 per 20 samples, minimum of one per batch of samples processed Criteria: Percent recovery must be within laboratory control limits Corrective Action: Flag data outside of limit	9060 Walkley-Black	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: Reanalyze if sample remaining. If not, flag data associated with unacceptable Matrix Spike

INORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES [1]	Method	RCRA (SW846) [2]
Total Organic Carbon (TOC) (cont'd)	Matrix Spike Duplicate	415.1 SM5310D	Frequency: 1 per 20 samples, minimum of one per batch of samples processed Criteria: Percent recovery must be within laboratory control limits Corrective Action: Flag data outside of limit	9060 Walkley-Black	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: Reanalyze if sample remaining. If not, flag data associated with unacceptable Matrix Spike Duplicate
	Duplicate	415.1 SM5310D	N/A	9060 Walkley-Black	Not Applicable
Total Organic Halides (TOX)	* Method Blank	450.1 (5)	Frequency: 1 with each set of 8 samples Criteria: Concentration less than reporting limit Corrective Action: Rerun all samples associated with unacceptable blank	9020B	Frequency: Run in duplicate between each group of 8 analytical determinations Criteria: Concentration less than reporting limit or less than 2 X MDL or RL whichever is lower Corrective Action: Rerun all samples associated with unacceptable blank
	Laboratory Control Sample	450.1 (5)	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery of analyte must be within laboratory control limits Corrective Action: Rerun all samples associated with unacceptable LCS (ICV)	9020B	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery of analyte must be within 90-110% Corrective Action: Rerun all samples associated with unacceptable LCS (ICV)

INORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES [1]	Method	RCRA (SW846) [2]
Total Organic Halides (TOX) (cont'd)	Matrix Spike	450.1 (5)	Frequency: 1 per 20 samples, minimum of one per batch of samples processed Criteria: Must be within laboratory control limits Corrective Action: Reanalyze if sample remaining. If not, flag data with unacceptable Matrix Spike	9020B	Frequency: 1 per batch of 10 samples Criteria: Must be within laboratory control limits Corrective Action: Flag data associated with unacceptable Matrix Spike SOP NO. CORP-WC-0001
	Matrix Spike Duplicate	450.1(5)	Frequency: 1 per 20 samples, minimum of one per batch of samples processed Criteria: Must be within laboratory control limits Corrective Action: Reanalyze if sample remaining. If not, flag data with unacceptable Matrix Spike	9020B	N/A
	Duplicate	450.1(5)	**N/A**	9020B	N/A
Turbidity	* Method Blank	180.1	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration must be less than the reporting limit Corrective Action: Rerun all samples associated with unacceptable blank	—	N/A

INORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES [1]	Method	RCRA (SW846) [2]
Turbidity (cont'd)	Laboratory Control Sample	180.1	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: If not within laboratory control limits, rerun all associated samples	—	N/A
	Matrix Spike	180.1	N/A	—	N/A
	Matrix Spike Duplicate	180.1	N/A	—	N/A
	Duplicate	180.1	Frequency: 1 per 10 samples Criteria: Must be within laboratory QC limits Corrective Action: Flag data outside of limit Not Applicable.	—	N/A
Mercury by CVAA & CVAFS	* Method Blank	200 series 1631E	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration less than reporting limit Corrective Action: Rerun all samples associated with unacceptable blank, unless the blank is above RL, and samples are ND.	7000A series	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration less than reporting limit Corrective Action: Rerun all samples associated with unacceptable blank, unless the blank is above RL and samples are ND.

INORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES [1]	Method	RCRA (SW846) [2]
Mercury by CVAA & CVAFS (Cont'd)	Laboratory Control Sample	200 series 1631E	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: percent recovery of analyte must be within ± 20 %. 1631E is +/- 23% Corrective Action: Rerun all samples associated with unacceptable LCS, unless samples are ND, results are reported.	7000A series	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: percent recovery of analyte must be within ± 20 % Corrective Action: Rerun all samples associated with unacceptable LCS, unless MT-0003. Exception: If samples are ND, results are reported.
	Matrix Spike	200 series 1631E	Frequency: with each batch of samples processed not to exceed 20 samples. 1631E frequency is 1 in 10 samples, 71-125% Criteria: Recovery should be within 75-125 % Corrective Action: Flag data associated with unacceptable MS.	7000A series	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Recovery should be within 75-125 % Corrective Action: Flag data associated with unacceptable MS.
	Matrix Spike Duplicate	200 series 1631E	Frequency: 1 with each batch of samples processed not to exceed 20 samples. 1631E frequency is 1 in 10 samples, 71-125% RPD 24% Criteria: Recovery should be within 75-125 % , RPD(3) should be within 20% Corrective Action: Flag data associated with unacceptable MSD	7000A series	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Recovery should be within 75-125 % , RPD(3) should be within 20% Corrective Action: Flag data associated with unacceptable MSD

INORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES [1]	Method	RCRA (SW846) [2]
Mercury by CVAA & CVAFS (Cont'd)	Duplicate	200 series 1631E	N/A	7000A series	N/A
ICP Metals	* Method Blank	200.7 200.8	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration less than reporting limit. Concentration less than reporting with the exception of lab common contaminants. Sample results <RL are also valid. Corrective Action: Rerun all samples associated with unacceptable blank unless the blank is above RL, and samples are ND. SOP NC-MT-012	6010B 6020	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration less than reporting limit. Concentration less than reporting with the exception of lab common contaminants. Sample results <RL are also valid. Corrective Action: Rerun all samples associated with unacceptable blank unless the blank is above RL, and samples are ND. SOP NC-MT-012
	Laboratory Control Sample	200.7 200.8	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: percent recovery of analyte must be ± 85-115%. If LCS is biased high and samples are <RL, the results are valid. Corrective Action: Rerun all samples associated with unacceptable LCS SOP NC-MT-012	6010B 6020	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: percent recovery of analyte must be ± 20 %. If LCS is biased high and samples are <RL, the results are valid. Corrective Action: Rerun all samples associated with unacceptable LCS SOP NC-MT-012. If samples are ND, results are reported.

INORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES [1]	Method	RCRA (SW846) [2]
ICP Metals (Cont'd)	Matrix Spike	200.7 200.8	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Limits for percent recovery are 75-125% Corrective Action: Flag data associated with unacceptable matrix spike SOP NC-MT-012	6010B 6020	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Limits for percent recovery are 75-125% Corrective Action: Flag data associated with unacceptable matrix spike SOP NC-MT-012
	Matrix Spike Duplicate	200.7 200.8	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Limits for percent recovery are 75-125%, RPD(3) must be within 20% Corrective Action: Flag data associated with unacceptable matrix spike SOP NC-MT-012	6010B 6020	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Limits for percent recovery are 75-125%, RPD(3) must be within 20% Corrective Action: Flag data associated with unacceptable matrix spike SOP NC-MT-012
	Duplicate	200.7 200.8	Not Applicable	6010B 6020	Not Applicable
	Serial Dilution	200.7 200.8	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: 10% difference. 10% difference only applied if sample results are >50 times IDL. Corrective Action: Flag data associated with unacceptable serial dilution SOP NC-MT-012	6010B 6020	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: 10% difference. 10% difference only applied if sample results are >50 times IDL. Corrective Action: Flag data associated with unacceptable serial dilution SOP NC-MT-012

INORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Footnotes

[1] National Pollutant Discharge Elimination System

[2] Resource Conservation and Recovery Act, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, (SW-846), Third Edition, September 1986. Contains Final Update I (July 1992), Final Update IIA (August 1993), Final Update II (September 1994), Final Update IIB (January 1995), and Final Update III (December 1996).

[3] RPD-Relative Percent Difference

[4] Orthophosphate only

[5] Method not listed in 40 CFR Part 136. Method 300.0 is a proposed 40CFR method. Specific state and/or region approval is required for NPDES.

[6] Current promulgated method is a Guidance Method Only, SW-846, Final Update III, Rev.3, 12/96.

ORGANIC LABORATORY QUALITY CONTROL SAMPLES

Analysis	QC Sample	Method	NPDES [1]	Method	RCRA (SW846) [2]
Aromatic Volatiles by GC	* Method Blank	602	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration less than reporting limit Corrective Action: Rerun all samples associated with unacceptable blank	8021B	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration less than reporting limit Corrective Action: Rerun all samples associated with unacceptable blank
	Laboratory Control Sample	602	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: percent recovery must be within acceptance limits given in method for each analyte Corrective Action: Rerun all samples associated with unacceptable LCS	8021B	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: percent recovery for each analyte must be within laboratory acceptance limits Corrective Action: Rerun all samples associated with unacceptable LCS
	Matrix Spike	602	Frequency: 1 per 10 samples from each site or 1 per month, whichever is more frequent Criteria: percent recovery for each analyte should be within advisory limits given in method Corrective Action: Flag data associated with unacceptable Matrix Spike	8021B	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: percent recovery for each analyte should be within laboratory acceptance limits Corrective Action: Flag data associated with unacceptable Matrix Spike

ORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES [1]	Method	RCRA (SW846) [2]
Aromatic Volatiles by GC (Cont'd)	Matrix Spike Duplicate	602	N/A	8021B	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: percent recovery for each analyte should be within laboratory acceptance limits Corrective Action: Flag data associated with unacceptable Matrix Spike
	Duplicate	602	N/A	8021B	N/A
	Surrogates	602	Surrogates spiked into method blank and all samples (QC included) Method Blank Criteria and LCS: All surrogates must be within laboratory established control limits before sample analysis may proceed. Sample Criteria: Re-extract samples or flag sample data not meeting surrogate criteria	8021B	Surrogates spiked into method blank and all samples (QC included) Method Blank Criteria and LCS: All surrogates must be within laboratory established control limits before sample analysis may proceed. Sample Criteria: Reprepare and reanalyze samples or flag sample data not meeting surrogate criteria
	Internal Standards	602	Optional: Internal standards are added to the method blank and all samples (QC included).	8021B	Optional: Internal standards are added to the method blank and all samples (QC included). If used, same compounds as used for surrogates may be appropriate.

ORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES [1]	Method	RCRA (SW846) [2]
Halogenated Volatiles by GC	* Method Blank	--	N/A	8021B	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration less than reporting limit Corrective Action: Rerun all samples associated with unacceptable blank
	Laboratory Control Sample	--	N/A	8021B	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: percent recovery for each analyte must be within laboratory acceptance limits Corrective Action: Rerun all samples associated with unacceptable LCS
	Matrix Spike	--	N/A	8021B	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: percent recovery for each analyte should be within laboratory acceptance limits Corrective Action: Flag data associated with unacceptable Matrix Spike
	Matrix Spike Duplicate	--	N/A	8021B	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: percent recovery for each analyte should be within laboratory acceptance limits Corrective Action: Flag data associated with unacceptable Matrix Spike

ORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES [1]	Method	RCRA (SW846) [2]
Halogenated Volatiles by GC (Cont'd)	Duplicate	--	N/A	8021B	N/A
	Surrogates	--	N/A	8021B	Surrogates spiked into method blank and all samples (QC included) Method Blank Criteria and LCS: All surrogates must be within laboratory established control limits before sample analysis may proceed. Sample Criteria: Reprepare and reanalyze samples or flag sample data not meeting surrogate criteria.
	Internal Standards	--	N/A	8021B	Optional: Internal standards are added to the method blank and all samples (QC included). If used, same compounds as used for surrogates may be appropriate.

ORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES [1]	Method	RCRA (SW846) [2]
Herbicides	Laboratory Control Sample	615 [3]	Frequency: 1 with each extraction batch of samples not to exceed 20 samples Criteria: Percent recovery must be within acceptance limits given in method for each analyte Corrective Action: Re-extract all samples associated with unacceptable LCS	8151A	Frequency: 1 with each extraction batch of samples not to exceed 20 samples Criteria: Percent recovery for each analyte must be within laboratory acceptance limits Corrective Action: Re-extract and reanalyze all samples associated with unacceptable LCS
	Matrix Spike	615 [3]	Frequency: 1 per 10 samples from each site or 1 per month, whichever is more frequent Criteria: Percent recovery for each analyte should be within advisory limits given in method Corrective Action: Flag data associated with unacceptable Matrix Spike	8151A	Frequency: 1 with each extraction batch of samples not to exceed 20 samples Criteria: Percent recovery for each analyte should be within laboratory acceptance limits Corrective Action: Flag data associated with unacceptable Matrix Spike
	Matrix Spike Duplicate	615 [3]	N/A	8151A	Frequency: 1 with each extraction batch of samples not to exceed 20 samples Criteria: percent recovery for each analyte should be within laboratory control limits Corrective Action: Flag data associated with unacceptable matrix spike sample

ORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES [1]	Method	RCRA (SW846) [2]
Herbicides (cont'd)	Duplicate	615 [3]	N/A	8151A	N/A
	Surrogates	615 [3]	N/A	8151A	Surrogates spiked into method blank and all samples (QC included) Method Blank Criteria and LCS: All surrogates must fall within laboratory established control limits before sample analysis may proceed. Sample Criteria: Re-extract and reanalyze samples or flag sample data not meeting surrogate criteria
	Internal Standards	615 [3]	N/A	8151A	Optional
Pesticides/ PCBs	* Method Blank	608	Frequency: 1 with each extraction batch of samples not to exceed 20 samples Criteria: Concentration less than reporting limit Corrective Action: Rerun all samples associated with unacceptable blank	8081A 8082	Frequency: 1 with each extraction batch of samples not to exceed 20 samples Criteria: Concentration less than reporting limit Corrective Action: Reprepare and reanalyze all samples associated with unacceptable blank

ORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES [1]	Method	RCRA (SW846) [2]
Pesticides/ PCBs (Cont'd)	Laboratory Control Sample	608	Frequency: 1 with each extraction batch of samples not to exceed 20 samples Criteria: percent recovery must be within acceptance limits given in method for each analyte Corrective Action: Rerun all samples associated with unacceptable LCS	8081A 8082	Frequency: 1 with each extraction batch of samples not to exceed 20 samples Criteria: percent recovery for each analyte must be within laboratory acceptance limits Corrective Action: Rerun all samples associated with unacceptable LCS
	Matrix Spike	608	Frequency: 1 per 10 samples from each site or 1 per month, whichever is more frequent Criteria: percent recovery for each analyte should be within advisory limits given in method Corrective Action: Flag data associated with unacceptable Matrix Spike	8081A 8082	Frequency: 1 with each extraction batch of samples not to exceed 20 samples Criteria: percent recovery for each analyte should be within laboratory acceptance limits Corrective Action: Flag data associated with unacceptable Matrix Spike

ORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES [1]	Method	RCRA (SW846) [2]
Pesticides/ PCBs (cont'd)	Matrix Spike Duplicate	608	N/A	8081A 8082	Frequency: 1 with each extraction batch of samples not to exceed 20 samples Criteria: percent recovery for each analyte should be within laboratory acceptance limits Corrective Action: Flag data associated with unacceptable Matrix Spike
	Duplicate	608	N/A	8081A 8082	N/A
	Surrogates	608	Not specified in method	8081A 8082	Surrogates spiked into method blank and all samples (QC included) Method Blank Criteria and LCS: Results must fall within laboratory established control limits Sample Criteria: Re-extract and reanalyze samples or flag sample data not meeting surrogate criteria
Petroleum Hydro-carbons	* Method Blank	1664A	Frequency: 1 with each preparation batch Criteria: Concentration must be less than the reporting limit Corrective Action: Rerun all samples associated with unacceptable blank	9071B	Frequency: 1 with each preparation batch Criteria: Concentration must be less than the reporting limit Corrective Action: Rerun all samples associated with unacceptable blank

ORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES [1]	Method	RCRA (SW846) [2]
Petroleum Hydro-carbons (Cont'd)	Laboratory Control Sample	1664A	Frequency: 1 with each analytical batch Criteria: Waters - See limits in SOP, NC-WC-0084 Soils - Percent recovery must be within laboratory control limits Corrective Action: Rerun all samples associated with unacceptable LCS	9071B	Frequency: 1 with each analytical batch Criteria: Waters - See limits in SOP, NC-WC-0084 Soils - Percent recovery must be within laboratory control limits Corrective Action: Rerun all samples associated with unacceptable LCS
	Matrix Spike	1664A	Frequency: 1 with every 10 samples per site Criteria: See percent recovery limits in SOP, NC-WC-0084 Corrective Action: See SOP, NC-WC-0084	9071B	Frequency: 1 with every 10 samples per site Criteria: See percent recovery limits in SOP, NC-WC-0084 Corrective Action: See SOP, NC-WC-0084
	Matrix Spike Duplicate	1664A	Frequency: 1 with every 20 samples per site Criteria: See percent recovery and RPD limits in SOP, NC-WC-0084 Corrective Action: See NC-WC-0084	9071B	Frequency: 1 with every 10 samples per site Criteria: See percent recovery and RPD limits in SOP, NC-WC-0084 Corrective Action: See NC-WC-0084
	Duplicate	1664A	N/A	9071B	N/A

ORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES [1]	Method	RCRA (SW846) [2]
Purgeable Halocarbons by GC	* Method Blank	601	Frequency: 1 with each extraction batch of samples not to exceed 20 samples Criteria: Concentration less than reporting limit Corrective Action: Rerun all samples associated with unacceptable blank	8021B	Frequency: 1 with each extraction batch of samples not to exceed 20 samples Criteria: Concentration less than reporting limit Corrective Action: Rerun all samples associated with unacceptable blank
	Laboratory Control Sample	601	Frequency: 1 with each extraction batch of samples not to exceed 20 samples Criteria: percent recovery must be within acceptance limits given in method for each analyte Corrective Action: Rerun all samples associated with unacceptable LCS	8021B	Frequency: 1 with each extraction batch of samples not to exceed 20 samples Criteria: percent recovery for each analyte must be within laboratory acceptance limits Corrective Action: Rerun all samples associated with unacceptable LCS
	Matrix Spike	601	Frequency: 1 per 10 samples from each site or 1 per month, whichever is more frequent Criteria: percent recovery for each analyte should be within advisory limits given in method Corrective Action: Flag data associated with unacceptable Matrix Spike	8021B	Frequency: 1 with each extraction batch of samples not to exceed 20 samples Criteria: percent recovery for each analyte should be within laboratory acceptance limits Corrective Action: Flag data associated with unacceptable Matrix Spike

ORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES [1]	Method	RCRA (SW846) [2]
Purgeable Halocarbons by GC (Cont'd)	Matrix Spike Duplicate	601	N/A	8021B	Frequency: 1 with each extraction batch of samples not to exceed 20 samples Criteria: percent recovery for each analyte should be within laboratory acceptance limits Corrective Action: Flag data associated with unacceptable Matrix Spike
	Duplicate	601	N/A	8021B	N/A
	Surrogates	601	Surrogates spiked into method blank and all samples (QC included) Method Blank Criteria and LCS: All surrogates must be within laboratory established control limits before sample analysis may proceed. Sample Criteria: Re-extract samples or flag sample data not meeting surrogate criteria	8021B	Surrogates spiked into method blank and all samples (QC included) Method Blank Criteria and LCS: All surrogates must be within laboratory established control limits before sample analysis may proceed. Sample Criteria: Re-extract and reanalyze samples or flag sample data not meeting surrogate criteria
	Internal Standards	601	Optional: Internal standards are added to the method blank and all samples (QC included). If used, same compounds as used for surrogates may be appropriate.	8021B	Optional: Internal standards are added to the method blank and all samples (QC included). If used, same compounds as used for surrogates may be appropriate.

ORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES [1]	Method	RCRA (SW846) [2]
Semivolatiles	Matrix Spike	625	Frequency: 1 with each extraction batch of samples not to exceed 20 samples Criteria: percent recovery for each analyte should be within advisory limits given in method Corrective Action: Flag data associated with unacceptable Matrix Spike	8270C	Frequency: 1 with each extraction batch of samples not to exceed 20 samples Criteria: percent recovery for each analyte should be within laboratory acceptance limits Corrective Action: Flag data associated with unacceptable Matrix Spike
	Matrix Spike Duplicate	625	N/A	8270C	Frequency: 1 with each extraction batch of samples not to exceed 20 samples Criteria: percent recovery for each analyte should be within laboratory acceptance limits Corrective Action: Flag data associated with unacceptable Matrix Spike

ORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES [1]	Method	RCRA (SW846) [2]
Semivolatiles (Cont'd)	Duplicate	625	N/A	8270C	N/A
	Surrogates	625	Surrogates spiked into method blank and all samples (QC included) Method Blank and LCS Criteria: All surrogates must be in control before sample analysis may proceed. One surrogate per fraction may exceed control limits if greater than 10% recovery. Sample Criteria: Re-extract samples or flag sample data not meeting surrogate criteria	8270C	Surrogates spiked into method blank and all samples (QC included) Method Blank and LCS Criteria: All surrogates must be in control before sample analysis may proceed. One surrogate per fraction may exceed control limits if greater than 10% recovery. Sample Criteria: Re-extract and reanalyze samples or flag sample data not meeting surrogate criteria
	Internal Standards	625	Frequency: Internal standards spiked into method blank and all samples (QC included) Criteria: All internal standard recoveries must be within laboratory control limits Corrective Action: Flag sample data not meeting internal standard recovery requirements	8270C	Internal Standards are added to all samples (QC samples included). Internal standard area of daily standard must be within 50% to 200% of the response in the mid level of the initial calibration standard. The retention time (RT) for any internal standard (IS) in the continuing calibration must not exceed ± 0.5 minutes from mid level initial calibration standard IS RT.

ORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES [1]	Method	RCRA (SW846) [2]
Volatiles by GC/MS	* Method Blank	624	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration less than reporting limit Corrective Action: Rerun all samples associated with unacceptable blank	8260B	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration less than reporting limit Corrective Action: Rerun all samples associated with unacceptable blank
	Laboratory Control Sample	624	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: percent recovery for each analyte should be within advisory limits given in method Corrective Action: Flag data associated with unacceptable Matrix Spike	8260B	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: percent recovery for each analyte must be within laboratory acceptance limits Corrective Action: Rerun all samples associated with unacceptable LCS

ORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES [1]	Method	RCRA (SW846) [2]
Volatiles by GC/MS (Cont'd)	Matrix Spike	624	Frequency: 1 per ≤ 20 samples from each site or 1 per month, whichever is more frequent Criteria: percent recovery for each analyte should be within advisory limits given in method Corrective Action: Flag data associated with unacceptable Matrix Spike	8260B	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: percent recovery for each analyte should be within laboratory acceptance limits Corrective Action: Flag data associated with unacceptable Matrix Spike
	Matrix Spike Duplicate	624	N/A	8260B	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: percent recovery for each analyte should be within laboratory acceptance limits Corrective Action: Flag data associated with unacceptable Matrix Spike
	Duplicate	624	N/A	8260B	N/A

ORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES [1]	Method	RCRA (SW846) [2]
Volatiles by GC/MS (cont'd)	Surrogates	624	Surrogates spiked into Method Blank and all samples (QC included) Method Blank Criteria: All surrogates must be in control before sample analysis may proceed. Sample Criteria: Re-extract samples or flag sample data not meeting surrogate criteria	8260B	Surrogates spiked into Method Blank and all samples (QC included) Method Blank Criteria and LCS: All surrogates must be in control before sample analysis may proceed. Sample Criteria: Re-extract and reanalyze samples or flag sample data not meeting surrogate criteria
	Internal Standards	624	Frequency: Internal standards spiked into method blank and all samples (QC included) Criteria: All internal standard recoveries must be within laboratory control limits Corrective Action: Flag sample data not meeting internal standard recovery requirements	8260B	Internal Standards are added to all samples (QC samples included). Internal standard area of daily standard must be within 50% to 200% of the response in the mid level of the initial calibration standard. The retention time (RT) for any internal standard (IS) in the continuing calibration must not exceed ± 0.5 minutes from mid level initial calibration standard IS RT.

Footnotes

[1] National Pollutant Discharge Elimination System

[2] Resource Conservation and Recovery Act, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, (SW-846), Third Edition, September 1986. Contains Final Update I (July 1992), Final Update IIA (August 1993), Final Update II (September 1994), Final Update IIB (January 1995), and Final Update III (December 1996)

[3] Method not listed in 40 CFR Part 136

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SECTION 13

PREVENTIVE ACTION
(*NELAC 5.4.11)*

13.1 OVERVIEW

The laboratory's preventive action programs improve, or eliminate potential causes of nonconforming product and/or nonconformance to the quality system. This preventive action process is a proactive continuous process improvement activity that can be initiated through feedback from clients, employees, business providers, and affiliates. The QA Department has the overall responsibility to ensure the preventive action process is in place, and that relevant information on actions is submitted for management review.

Dedicating resources to an effective preventive action system emphasizes the laboratory's commitment to its Quality Program. It is beneficial to identify and address negative trends before they develop into complaints, problems and corrective actions. Additionally, customer service and satisfaction can be improved through continuous improvements to laboratory systems.

Opportunities for improvement may be discovered during management reviews, the QA Metrics Report, internal or external audits, proficiency testing performance, client complaints, staff observation, etc.

The monthly QA Metrics Report shows performance indicators in all areas of the quality system. These areas include revised reports, corrective actions, audit findings, internal auditing and data authenticity audits, client complaints, PT samples, holding time violations, SOPs, Ethics training, etc. These metrics are used to help evaluate quality system performance on an ongoing basis and provide a tool for identifying areas for improvement.

The laboratory's corrective action process (Section 13) is integral to implementation of preventive actions. A critical piece of the corrective action process is the implementation of actions to prevent further occurrence of a non-compliance event. Historical review of corrective action provides a valuable mechanism for identifying preventive action opportunities.

13.1.1 The following elements are part of a preventive action system:

- Identification of an opportunity for preventive action.
- Process for the preventive action.
- Define the measurements of the effectiveness of the process once undertaken.
- Execution of the preventive action.
- Evaluation of the plan using the defined measurements.
- Verification of the effectiveness of the preventive action.
- Close-out by documenting any permanent changes to the Quality System as a result of the Preventive Action. Documentation of Preventive Action is incorporated into the monthly QA reports, corrective action process, and management review.

13.1.2 Any Preventive Actions undertaken or attempted must be taken into account during the Annual Management Review (Section 16). A highly detailed recap is not required; a simple recount of success and failure within the preventive action program will provide management a measure for evaluation.

13.2 MANAGEMENT OF CHANGE

The Management of Change process is designed to manage significant events and changes that occur within the laboratory. Through these procedures, the potential risks inherent with a new event or change are identified and evaluated. The risks are minimized or eliminated through pre-planning and the development of preventive measures. The types of changes covered under this system include: Facility Changes, Major Accreditation Changes, Addition or Deletion to Division's Capabilities or Instrumentation, Key Personnel Changes, Laboratory Information Management System (LIMS) changes. This process is discussed in further detail in SOP CA-Q-S-003, Management of Change.

SECTION 14

CONTROL OF RECORDS
(NELAC 5.4.12)

The laboratory maintains a record system appropriate to its needs and that complies with applicable standards or regulations as required. The system produces unequivocal, accurate records that document all laboratory activities. The laboratory retains all original observations, calculations and derived data, calibration records and a copy of the analytical report for a minimum of five years after it has been issued.

14.1 OVERVIEW

The laboratory has established procedures for identification, collection, indexing, access, filing, storage, maintenance and disposal of quality and technical records. A record index is listed in Table 14-1. Quality records are maintained by the QA Department in a database which is backed up as part of the regular network backup. Records are of two types--either electronic or hard-copy paper formats depending on whether the record is computer or hand generated (some records may be in both formats). Technical records are maintained by the Records Manager.

Table 14-1. Records Index

Type of Record	Retention	Disposition
General Laboratory Documents		
Instrument output	5 yrs from project completion	Shred or burn
Quality control data	5 yrs from project completion*	Shred or burn
Field sample data	5 yrs from project completion	Shred or burn
Final analytical reports	5 yrs from project completion	Shred or burn
Instrument logbooks	5 yrs from last entry	Shred or burn
Equipment monitoring & maintenance records	5 yrs from last entry	Shred or burn
Instrument calibration records	5 yrs from last entry	Shred or burn
Standard preparation logs	5 yrs from last entry	Shred or burn
Standards certificates	5 yrs from last entry	Shred or burn
Measurement & test equipment logs (e.g., refrig., balances, etc.)	5 yrs from last entry	Shred or burn
Method & instrument validation records	5 yrs from last entry	Shred or burn
Instrument manuals	Retain until superseded	Trash
Project management files	5 yrs from date of archival	Shred or burn
Quotes & proposals	2 yrs from date of expiration	Shred or burn
LQM, policies, & SOPs	5 yrs from date of archiving	Shred or burn

Type of Record	Retention	Disposition
Analyst demonstrations of proficiency	5 yrs from date of archival	Shred or burn
Quality assurance audits	5 yrs from last entry	Shred or burn
Certifications & approvals	5 yrs from last entry	Shred or burn
Employee signature list	5 yrs from date of archival	Shred or burn
MDL Studies	5 yrs from last entry	Shred or burn
Performance testing studies	5 yrs from last entry	Shred or burn
QA reports to management	5 yrs from last entry	Shred or burn
Quality control charts	5 yrs from last entry	Shred or burn
Environment, Health and Safety Records		
Medical records	Retain while active & 30 yrs from last entry	Shred or burn
Employee exposure & monitoring records	Retain while active & 30 yrs from last entry	Shred or burn
Workers compensation files & first report of injury	Retain while active & 30 yrs from last entry	Shred or burn
Accident logs (OSHA Form 200)	5 yrs from last entry	Shred or burn
Accident reports	5 yrs from last entry	Shred or burn
Environmental permits	5 yrs from last entry	Shred or burn
Environmental management, e.g., discharge reports	5 yrs from last entry	Shred or burn
Health & safety audits	5 yrs from last entry	Shred or burn
Chemical Hygiene Plan	5yrs from archival	
Safety Inspections	5 yrs from last entry	Shred or burn
TLD exposure records	5 yrs from last entry	Shred or burn
EH&S training	5 yrs from last entry	Shred or burn
Accounting	See Accounting and Controls Procedures Manual	
Administrative		
Personnel records (not including medical or disability records)	7 years from last entry	Shred or burn

* Exceptions listed in Table 14-2.

All records are stored and retained in such a way that they are secure and readily retrievable at the laboratory facility that provides a suitable environment to prevent damage or deterioration and to prevent loss**.** All records must be protected against fire, theft, loss, environmental deterioration, and vermin. In the case of electronic records and electronic or magnetic sources, storage media are protected from deterioration caused by magnetic fields and/or electronic deterioration.

Access to the data is limited to laboratory and company employees. Records are maintained for a minimum of five years unless otherwise specified by a client or regulatory requirement.

For raw data and project records, record retention must be calculated from the date the project report is issued. For other records, such as Controlled Documents, QA, or Administrative Records, the retention time is calculated from the date the record is formally retired. Records related to the programs listed in Table 14-2 have lengthier retention requirements and are subject to the requirements in Section 14.1.3.

14.1.1 Programs with Longer Retention Requirements

Some regulatory programs have longer record retention requirements than the standard record retention time. These are detailed in Table 14-2 with their retention requirements. In these cases, the longer retention requirement is enacted. If special instructions exist such that client data cannot be destroyed prior to notification of the client, the container or box containing that data is marked as to who to contact for authorization prior to destroying the data.

Note: For the Ohio VAP program the laboratory is required to notify Ohio EPA of its intent to dispose of any records.

Table 14-2. Special Record Retention Requirements

Program	Retention Requirement
Ohio – Drinking Water	10 years
Michigan Department of Environmental Quality – all environmental data	10 years
OSHA - 40 CFR Part 1910	30 years
TSCA - 40 CFR Part 792	10 years after publication of final test rule or negotiated test agreement and others as negotiated.
Ohio Voluntary Action Program	10 years

Note: Extended retention requirements must be noted with the archive documents or addressed in facility-specific records retention procedures.

14.1.2 The laboratory has procedures to protect and back-up records stored electronically and to prevent unauthorized access to or amendment of these records. All analytical data is maintained as hardcopy or in a secure readable electronic format. For analytical reports that are maintained as copies in PDF format, refer to Section 19.12.1 for more information.

14.1.3 The record-keeping system allows for historical reconstruction of all laboratory activities that produced the analytical data, as well as rapid recovery of historical data. (Records stored off site should be accessible within two days of a request for such records). The history of the sample from when the laboratory took possession of the samples must be readily understood through the documentation. This must include inter-laboratory transfers of samples and/or extracts.

- The records include the identity of personnel involved in sampling, sample receipt, preparation, or testing. All analytical work contains the initials (at least) of the personnel involved. The laboratory copy of the Chain-of-Custody is stored with the invoice and the Work Order sheet generated by LIMS. The Chain-of-Custody would indicate the name of the sampler. If any sampling notes are provided with a Work Order, they are kept with this package.

- All information relating to the laboratory facilities equipment, analytical test methods, and related laboratory activities, such as sample receipt, sample preparation, or data verification are documented.

- The record-keeping system facilitates the retrieval of all working files and archived records for inspection and verification purposes, e.g., set format for naming electronic files, set format for what is included with a given analytical data set. SOP NC-QA-019, Records Information Management, outlines this procedure. Instrument data is stored sequentially by instrument. A given day's analyses are maintained in the order of the analysis. Run logs are maintained for each instrument or method; a copy of each day's run long or instrument sequence is stored with the data to aid in re-constructing an analytical sequence. Where an analysis is performed without an instrument, bound logbooks or bench sheets are used to record and file data. Standard and reagent information is recorded in logbooks or entered into LIMS for each method as required.

- Changes to hardcopy records must follow the procedures outlined in Sections 12 and 19. Changes to electronic records in LIMS or instrument data are recorded in audit trails.

- The reason for a signature or initials on a document is clearly indicated in the records such as "Sampled by," "Prepared by," "Reviewed by", or "Analyzed by".

- All generated data except those that are generated by automated data collection systems, are recorded directly, promptly and legibly in permanent dark ink.

- Hard-copy data may be scanned into PDF format for record storage as long as the scanning process can be verified in order to ensure no data is lost, and the data files and storage media must be tested to verify the laboratory's ability to retrieve the information prior to the destruction of the hard-copy which was scanned.

- Also refer to Section 19.14.1, “Computer and Electronic Data Related Requirements”.

14.2 TECHNICAL AND ANALYTICAL RECORDS

14.2.1 The laboratory retains records of original observations, derived data and sufficient information to establish an audit trail, calibration records, staff records and a copy of each analytical report issued, for a minimum of five years unless otherwise specified by a client or regulatory requirement (refer to Section 15.1). The records for each analysis must contain sufficient information to enable the analysis to be repeated under conditions as close as possible to the original. The records must include the identity of laboratory personnel responsible for sample preparation, performance of each analysis and reviewing results.

14.2.2 Observations, data, and calculations are recorded in real-time at the time they are made and are identifiable to the specific task.

14.2.3 Changes to hardcopy records must follow the procedures outlined in Sections 12 and 19. Changes to electronic records in LIMS or instrument data are recorded in audit trails. The essential information to be associated with analysis, such as strip charts, tabular printouts, computer data files, analytical notebooks, and run logs, include:

- Laboratory sample ID code
- Date of analysis. Time of analysis is also required if the holding time is 72 hours or less, or when time-critical steps are included in the analysis (e.g., drying times, incubations, etc.); instrumental analyses have the date and time of analysis recorded as part of their general operations. Where a time critical step exists in an analysis, location for such a time is included as part of the documentation in a specific logbook or on a benchsheet.
- Instrumentation identification and instrument operating conditions/parameters. Operating conditions/parameters are typically recorded in instrument maintenance logs where available. Instrument logs may be in electronic format.
- Analysis type
- All manual calculations and manual integrations
- Analyst or operator initials/signature
- Sample preparation, including cleanup, separation protocols, incubation periods or subculture, ID codes, volumes, weights, instrument printouts, meter readings, calculations, reagents
- Test results
- Standard and reagent origin, receipt, preparation, and use
- Calibration criteria, frequency, and acceptance criteria
- Data and statistical calculations, review, confirmation, interpretation, assessment and reporting conventions
- Quality control protocols and assessment
- Electronic data security, software documentation and verification, software and hardware audits, backups, and records of any changes to automated data entries

- Method performance criteria including expected quality control requirements. These are indicated both in the LIMS and on specific analytical report formats**.**

14.3 LABORATORY SUPPORT ACTIVITIES

In addition to documenting all the above-mentioned activities, the following are retained QA records and project records (previous discussions in this section relate where and how these data are stored):

- All original raw data, whether hard-copy or electronic, for calibrations, samples and quality control measures, including analysts' work sheets and data output records (chromatograms, strip charts, and other instrument response readout records)
- A written description or reference to the specific test method used which includes a description of the specific computational steps used to translate parametric observations into a reportable analytical value
- Copies of final reports
- Archived SOPs
- Correspondence relating to laboratory activities for a specific project
- All Corrective Action reports, audits and audit responses
- Proficiency test results and raw data
- Results of data review, verification, and cross-checking procedures

14.3.1 Sample Handling Records

Records of all procedures to which a sample is subjected while in the possession of the laboratory are maintained. These include, but are not limited to, records pertaining to:

- Sample preservation including appropriateness of sample container and compliance with holding time requirement
- Sample identification, receipt, acceptance or rejection and login
- Sample storage and tracking including shipping receipts, sample transmittal / COC forms
- Procedures for the receipt and retention of samples, including all provisions necessary to protect the integrity of samples.

14.4 ADMINISTRATIVE RECORDS

The laboratory also maintains the administrative records in either electronic or hard-copy form Refer to Table 14-1.

14.5 RECORDS MANAGEMENT, STORAGE AND DISPOSAL

14.5.1 All records (including those pertaining to test equipment), certificates, and reports are safely stored, held secure, and in confidence to the client. Certification-related records are

available to the accrediting body upon request.

14.5.2 All information necessary for the historical reconstruction of data is maintained by the laboratory. Records that are stored only on electronic media must be supported by the hardware and software necessary for their retrieval.

14.5.3 Records that are stored or generated by computers or personal computers have hardcopy, write-protected backup copies, or an electronic audit trail controlling access**.**

14.5.4 The laboratory has a record management system for control of laboratory notebooks, instrument logbooks, standards logbooks, and records for data reduction, validation, storage, and reporting. Laboratory notebooks are issued on a per analysis basis, and are numbered sequentially.

14.5.5 Transfer of Ownership

In the event the laboratory transfers ownership or goes out of business, the laboratory shall ensure that the records are maintained or transferred according to client's instructions. Upon ownership transfer, record retention requirements shall be addressed in the ownership transfer agreement and the responsibility for maintaining archives is clearly established. In addition, in cases of bankruptcy, appropriate regulatory and state legal requirements concerning laboratory records must be followed. In the event of the closure of the laboratory, all records will revert to the control of the corporate headquarters. Should the entire company cease to exist, as much notice as possible will be given to clients and the accrediting bodies who have worked with the laboratory during the previous five years of such action.

14.5.6 Records Disposal

14.5.6.1 Records are removed from the archive and destroyed after five years, unless otherwise specified by a client or regulatory requirement. On a project-specific or program basis, clients may need to be notified prior to record destruction. Records are destroyed in a manner that ensures their confidentiality such as shredding, mutilation or incineration (refer to Tables 14-1 and 14-2).

14.5.6.2 Electronic copies of records must be destroyed by erasure or physically damaging off-line storage media so no records can be read.

14.5.6.3 If a third party records management company is hired to dispose of records, a "Certificate of Destruction" is required.

SECTION 15

AUDITS
(NELAC 5.4.13)

15.1 INTERNAL AUDITS

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Internal audits are performed to verify that laboratory operations comply with the requirements of the lab's quality system and with the external quality programs under which the laboratory operates. Audits are planned and organized by the QA staff. Personnel conducting the audits should be independent of the area being evaluated. Auditors will have sufficient authority, access to work areas, and organizational freedom necessary to observe all activities affecting quality and to report the assessments to laboratory management and when requested to corporate management.

Audits are conducted and documented as described in TestAmerica Corporate SOP CA-Q-S-004 on performing Internal Audits. The types and frequency of routine internal audits are shown in Table 16-1. Special or ad hoc assessments may be conducted as needed under the direction of the QA staff.

Table 15-1. Types of Internal Audits and Frequency

Description	Performed by	Frequency
Quality Systems	QA Department or Designee	All areas of the laboratory annually
QA Technical Audits - Evaluate raw data vs. final reports - Analyst integrity - Data authenticity	QA Department or Designee	All methods within a 2-year period, with at least 15% of methods every quarter
SOP Method Compliance	Group Leader	- All SOPs within a 2-year period - All new analysts or new analyst/methods within 3 months of IDOC
Special	QA Department or Designee	Surveillance or spot checks performed as needed
Performance Testing	Analysts with QA oversight	Two successful per year for each NELAC field of testing or as dictated by regulatory requirements

15.1.1 Annual Quality Systems Audit

An annual quality systems audit is required to ensure compliance to analytical methods and SOPs, laboratory Data Integrity and Ethics Policies, NELAC quality systems, client and state

requirements, and the effectiveness of the internal controls of the analytical process, including but not limited to, data review, quality controls, preventive action, and corrective action. The completeness of earlier corrective action is assessed. The audit is divided into modules for each operating or support area of the lab, and each module is comprehensive for a given area. The area audits may be done on a rotating schedule throughout the year to ensure adequate coverage of all areas. This schedule may change as situations in the laboratory warrant.

Note: Part of the quality systems audit relates to regulatory compliance. An assessment of the laboratory's compliance to regulatory requirements is performed by Corporate QA through monthly management reports, review of client and regulatory concerns and also through periodic on-site evaluations.

15.1.2 QA Technical Audits

QA technical audits are based on client projects, associated sample delivery groups, and the methods performed. Reported results are compared to raw data to verify the authenticity of results. The validity of calibrations and QC results are compared to data qualifiers, footnotes, and case narratives. Documentation is assessed by examining run logs and records of manual integrations. Manual calculations are checked. Where possible, MintMiner is used to identify unusual manipulations of the data deserving closer scrutiny. QA technical audits must include all methods within a two-year period.

15.1.3 SOP Method Compliance

Compliance of all SOPs with the source methods and compliance of the operational groups with the SOPs must be assessed by the Technical Director and the QA department at least every two years. The work of each newly hired analyst is assessed within three months of working independently, (e.g., completion of method IDOC). In addition, as analysts add methods to their capabilities, (new IDOC) reviews of the analyst work products must be performed within 3 months of completing the documented training.

15.1.4 Special Audits

Special audits are conducted on an as needed basis, generally as a follow up to specific issues such as client complaints, corrective actions, PT results, data audits, system audits, validation comments, regulatory audits or suspected ethical improprieties. Special audits are focused on a specific issue, and report format, distribution, and timeframes are designed to address the nature of the issue.

15.1.5 Performance Testing

The laboratory participates semi-annually in performance audits conducted through the analysis of PT samples provided by a third party. The laboratory generally participates in the following types of PT studies--nonpotable water and soil

It is TestAmerica's policy that PT samples be treated as typical samples in the production process. Furthermore, where PT samples present special or unique problems, in the regular production process they may need to be treated differently, as would any special or unique

request submitted by any client. The QA Manager must be consulted and in agreement with any decisions made to treat a PT sample differently due to some special circumstance.

Written responses to unacceptable PT results are required. In some cases it may be necessary for blind QC samples to be submitted to the laboratory to show a return to control.

15.2 EXTERNAL AUDITS

External audits are performed when certifying agencies or clients conduct on-site inspections or submit performance testing samples for analysis. It is TestAmerica's policy to cooperate fully with regulatory authorities and clients. The laboratory makes every effort to provide the auditors with access to personnel, documentation, and assistance. Laboratory group leaders are responsible for providing corrective actions to the QA Manager who coordinates the response for any deficiencies discovered during an external audit. Audit responses are due in the time allotted by the client or agency performing the audit. A copy of the audit report and the laboratory's Corrective Action plan must be forwarded to Corporate Quality.

The laboratory cooperates with clients and their representatives to monitor the laboratory's performance in relation to work performed for the client. The client may only view data and systems related directly to the client's work. All efforts are made to keep other client information confidential.

15.2.1 Confidential Business Information (CBI) Considerations

During on-site audits, auditors may come into possession of information claimed as business confidential. A business confidentiality claim is defined as "a claim or allegation that business information is entitled to confidential treatment for reasons of business confidentiality or a request for a determination that such information is entitled to such treatment." When information is claimed as business confidential, the laboratory must place on (or attach to) the information at the time it is submitted to the auditor, a cover sheet, stamped or typed legend or other suitable form of notice, employing language such as "trade secret", "proprietary" or "company confidential". Confidential portions of documents otherwise non-confidential must be clearly identified. CBI may be purged of references to client identity by the responsible laboratory official at the time of removal from the laboratory. However, sample identifiers may not be obscured from the information. Additional information regarding CBI can be found in within the 2003 NELAC standards.

15.3 AUDIT FINDINGS

Audit findings are documented using the Corrective Action process and database. The laboratory's Corrective Action responses for both types of audits may include action plans that could not be completed within a predefined timeframe. In these instances, a completion date must set and agreed to by Operations management and the QA Manager.

Developing and implementing Corrective Action to findings is the responsibility of the Department Manager where the finding originated. Findings that are not corrected by specified due dates are reported monthly to management in the QA monthly report. A copy of the audit report and the laboratory's Corrective Action plan must be forwarded to Corporate Quality.

If any audit finding casts doubt on the effectiveness of the operations or on the correctness or validity of the laboratory's test results, the laboratory must take timely corrective action, and must notify clients in writing if the investigations show that the laboratory results have been affected. Once corrective action is implemented, a follow-up audit is scheduled to ensure that the problem has been corrected.

Clients must be notified promptly in writing, of any event such as the identification of defective measuring or test equipment that casts doubt on the validity of results given in any test report or amendment to a test report. The investigation must begin within 24 hours of discovery of the problem and all efforts are made to notify the client within two weeks after the completion of the investigation.

SECTION 16

MANAGEMENT REVIEWS
(NELAC 5.4.14)

16.1 QUALITY ASSURANCE REPORT

A comprehensive QA Report must be prepared each month by the laboratory's QA Department and forwarded to the Laboratory Director/Manager and their Corporate Quality Director as well as the General Manager. All aspects of the QA system are reviewed to evaluate the suitability of policies and procedures. During the course of the year, the Laboratory Director/Manager, General Manager, or Corporate QA may request that additional information be added to the report.

On a monthly basis, Corporate QA compiles information from all the monthly laboratory reports. The Corporate Quality Directors prepare a report that includes a compilation of all metrics and notable information and concerns regarding the QA programs within the laboratories. The report also includes a listing of new regulations that may potentially impact the laboratories. This report is presented to the Senior Management Team and General Managers.

16.2 ANNUAL MANAGEMENT REVIEW

The Senior Lab Management Team (Laboratory Director, Technical Director, Operations Manager, QA Manager, HR Supervisor, I.T. Supervisor) conducts a review annually of its quality systems and LIMS to ensure its continuing suitability and effectiveness in meeting client and regulatory requirements and to introduce any necessary changes or improvements. It will also provide a platform for defining quality goals and objectives. Corporate Operations and Corporate QA personnel may be included in this meeting at the discretion of the Laboratory Director/Manager. The LIMS review consists of examining any audits, complaints or concerns that have been raised through the year that are related to the LIMS. The laboratory must summarize any critical findings that cannot be solved by the lab, and report them to Corporate IT.

The Management Systems Review (Corporate SOP CA-Q-S-008 and Work Instruction CA-Q-WI-020) uses information generated during the preceding year to assess the "big picture" by ensuring that routine actions taken and reviewed on a monthly basis are not components of larger systematic concerns. The monthly review should keep the quality systems current and effective; therefore, the annual review is a formal senior management process to review specific existing documentation. Significant issues from the following documentation are compiled or summarized by the QA Manager prior to the review meeting:

- Matters arising from the previous annual review
- Prior Monthly QA Reports issues
- Laboratory QA Metrics
- Review of report reissue requests
- Review of client feedback and complaints

- Issues arising from any prior management or staff meetings
- Minutes from prior Senior Lab Management Team meetings. Issues that may be raised from these meetings include:
  - Adequacy of staff, equipment and facility resources
  - Adequacy of policies and procedures
  - Future plans for resources and testing capability and capacity
- The annual internal double blind PT program sample performance (if performed)
- Compliance to the Ethics Policy and Data Integrity Plan, including any evidence/incidents of inappropriate actions or vulnerabilities related to data Integrity

A report is generated by the QA Manager and management. The report is distributed to the appropriate General Manager and Corporate Quality Director. The report includes, but is not limited to:

- The date of the review and the names and titles of participants
- A reference to the existing data quality related documents and topics that were reviewed
- Quality system or operational changes or improvements that will be made as a result of the review, e.g., an implementation schedule including assigned responsibilities for the changes (Action Table)

Changes to the quality systems requiring update to the laboratory QA Manual must be included in the next revision of the QA Manual.

16.3 POTENTIAL INTEGRITY RELATED MANAGERIAL REVIEWS

Potential integrity issues (data or business related) must be handled and reviewed in a confidential manner until such time as a follow-up evaluation, full investigation, or other appropriate actions have been completed and issues clarified. TestAmerica's Corporate Data Investigation/ Recall SOP CA-L-S-001 must be followed. All investigations that result in finding inappropriate activity are documented and include any disciplinary actions involved, corrective actions taken, and all appropriate notifications of clients.

TestAmerica's COO, VP of Client & Technical Services, General Managers and Corporate Quality Directors receive a monthly report from the Director of Quality & Client Advocacy summarizing any current data integrity or data recall investigations. The General Managers are also made aware of progress on these issues for their specific labs.

SECTION 17

PERSONNEL
(NELAC 5.5.2)

17.1 OVERVIEW

The laboratory's management believes that its highly qualified and professional staff is the single most important aspect in assuring a high level of data quality and service. The staff consists of professionals and support personnel as outlined in the organization chart in Figure 4-1.

All personnel must demonstrate competence in the areas where they have responsibility. Any staff that is undergoing training must have appropriate supervision until they have demonstrated their ability to perform their job function on their own. Staff must be qualified for their tasks based on appropriate education, training, experience and/or demonstrated skills as required.

The laboratory employs sufficient personnel with the necessary education, training, technical knowledge and experience for their assigned responsibilities.

All personnel are responsible for complying with all QA/QC requirements that pertain to the laboratory and their area of responsibility. Each staff member must have a combination of experience and education to adequately demonstrate a specific knowledge of their particular area of responsibility. Technical staff must also have a general knowledge of lab operations, test methods, QA/QC procedures and records management.

Laboratory management is responsible for formulating goals for lab staff with respect to education, training and skills and ensuring that the laboratory has a policy and procedures for identifying training needs and providing training of personnel. The training must be relevant to the present and anticipated responsibilities of the lab staff.

The laboratory only uses personnel that are employed by or under contract to, the laboratory. Contracted personnel, when used, must meet competency standards of the laboratory and work in accordance to the laboratory's quality system.

17.2 EDUCATION AND EXPERIENCE REQUIREMENTS FOR TECHNICAL PERSONNEL

The laboratory makes every effort to hire analytical staff that posses a college degree (AA, BA, BS) in an applied science with some chemistry in the curriculum. Exceptions can be made based upon the individual's experience and ability to learn. There are competent analysts and technicians in the industry who have not earned a college degree. Selection of qualified candidates for laboratory employment begins with documentation of minimum education, training, and experience prerequisites needed to perform the prescribed task. Minimum education and training requirements for TestAmerica employees are outlined in job descriptions and are generally summarized for analytical staff in the table below.

The laboratory maintains job descriptions for all personnel who manage, perform or verify work affecting the quality of the environmental testing the laboratory performs. Job Descriptions are located on the TestAmerica intranet "Human Resources" web-page (also see Section 4 for position descriptions/responsibilities).

Experience and specialized training are occasionally accepted in lieu of a college degree (basic lab skills such as using a balance, colony counting, aseptic or quantitation techniques, etc. are also considered

As a general rule for analytical staff:

Specialty	Education	Experience
Extractions, Digestions, some electrode methods (pH, DO, Redox, etc.), or Titrimetric and Gravimetric Analyses	H.S. Diploma	On the job training (OJT)
CVAA, FLAA, Single component or short list Chromatography (e.g., Fuels, BTEX-GC, IC	A college degree in an applied science or 2 years of college and at least one year of college chemistry	Or 2 years prior analytical experience is required
ICP, ICPMS, Long List or complex chromatography (e.g., Pesticides, PCB, Herbicides, HPLC, etc.), GCMS	A college degree in an applied science or 2 years of college chemistry	Or 5 years of prior analytical experience
Spectra Interpretation	A college degree in an applied science or 2 years of college chemistry	And 2 years relevant experience. Or 5 years of prior analytical experience
Technical Directors/Department Managers – General	Bachelors Degree in an applied science or engineering with 24 semester hours in chemistry An advanced (MS, PhD.) degree may substitute for one year of experience	And 2 years experience in environmental analysis of representative analytes for which they will oversee
Technical Director – Wet Chem only (no advanced instrumentation)	Associate degree in an applied science or engineering or 2 years of college with 16 semester hours in chemistry	And 2 years relevant experience

When an analyst does not meet these requirements, they can perform a task under the direct supervision of a qualified analyst, peer reviewer or Department Manager, and are considered an analyst in training. The person supervising an analyst in training is accountable for the quality of the analytical data and must review and approve data and associated corrective actions.

17.3 TRAINING

The laboratory is committed to furthering the professional and technical development of employees at all levels.

Orientation to the laboratory's policies and procedures, in-house method training, and employee attendance at outside training courses and conferences all contribute toward employee proficiency. Below are examples of various areas of required employee training:

Required Training	Time Frame*	Employee Type
New Hire Orientation	Immediately	All
Environmental Health & Safety Orientation	Prior to lab work	All
Environmental Health & Safety Orientation Follow-up Test	30-60 days after hire	All
Environmental Health & Safety Training	Refer to EH&S Manual	All
Ethics – New Hires	1 week of hire	All
Ethics - Comprehensive	90 days of hire	All
Data Integrity	30 days of hire	Technical and PMs
Quality Assurance	90 days of hire	All
Ethics – Comprehensive Refresher	Annually	All
Initial Demonstration of Capability (DOC)	Prior to unsupervised method performance	Technical

The laboratory maintains records of relevant authorization/competence, education, professional qualifications, training, skills and experience of technical personnel (including contracted personnel) as well as the date that approval/authorization was given. These records are kept on file at the laboratory. Also refer to "Demonstration of Capability" in Section 19.

The training of technical staff is kept up to date by:

- Each employee must have documentation in their training file that they have read, understood and agreed to follow the most recent version of the laboratory QA Manual and SOPs in their area of responsibility. This documentation is updated as SOPs are updated.
- Documentation from any training courses or workshops on specific equipment, analytical techniques or other relevant topics are maintained in the employee's training file.
- Documentation of proficiency (refer to Section 19)
- An Ethics Agreement signed by each staff member (renewed each year) and evidence of annual ethics training
- A Confidentiality Agreement signed by each staff member signed at the time of employment
- Human Resources maintains documentation and attestation forms on employment status & records; benefit programs; timekeeping/payroll; and employee conduct, e.g., ethics. This information is maintained in the employee's secured personnel file.

Evidence of successful training could include such items as:

- Adequate documentation of training within operational areas, including one-on-one technical training for individual technologies, and particularly for people cross-trained.
- Analysts' knowledge to refer to QA Manual for quality issues
- Analysts following SOPs, i.e., practice matches SOPs
- Analysts regularly communicate to group leaders and QA if SOPs need revision rather than waiting for auditors to find problems.

Further details of the laboratory's training program are described in the Laboratory Training SOP NC-QA-028, Employee Orientation and Training.

17.4 DATA INTEGRITY AND ETHICS TRAINING PROGRAM

Establishing and maintaining a high ethical standard is an important element of a Quality System. Ethics and data integrity training is integral to the success of TestAmerica and is provided for each employee at TestAmerica**.** It is a formal part of the initial employee orientation within one week of hire followed by technical data integrity training within 30 days, comprehensive training within 90 days, and annual refresher for all employees. Senior management at each facility performs the Ethics training for their staff.

In order to ensure that all personnel understand the importance TestAmerica places on maintaining high ethical standards at all times, TestAmerica has established a Corporate Ethics Policy (CA-L-P-001) and an Ethics Statement. All initial and annual training is documented by signature on the signed Ethics Statement/Agreement demonstrating that the employee has participated in the training and understands their obligations related to ethical behavior and data integrity.

Violations of this Ethics Policy will not be tolerated. Employees who violate this policy will be subject to disciplinary actions up to and including termination. Criminal violations may also be referred to the Government for prosecution. In addition, such actions could jeopardize TestAmerica's ability to do work on Government contracts; and for that reason, TestAmerica has a zero tolerance approach to such violations.

Employees are trained as to the legal and environmental repercussions that result from data misrepresentation. Key topics covered in the presentation include:

- Organizational mission and its relationship to the critical need for honesty and full disclosure in all analytical reporting
- Ethics Policy
- How and when to report ethical/data integrity issues. Confidential reporting.
- Record keeping
- Discussion regarding data integrity procedures

- Specific examples of breaches of ethical behavior--peak shaving, altering data or computer clocks, improper macros, etc., accepting/offering kickbacks, illegal accounting practices, unfair competition/collusion
- Internal monitoring. Investigations and data recalls
- Consequences for infractions including potential for immediate termination, debarment, or criminal prosecution
- Importance of proper written narration / data qualification by the analyst and project manager with respect to those cases where the data may still be usable but are in one sense or another partially deficient

Additionally, a Data Integrity Hotline (1-800-736-9407) is maintained by TestAmerica and administered by the Corporate Quality Department.

SECTION 18

ACCOMMODATIONS AND ENVIRONMENTAL CONDITIONS
(NELAC 5.5.3)

18.1 OVERVIEW

The laboratory is an 54,440 sq. ft. secure laboratory facility with controlled access and designed to accommodate an efficient workflow and to provide a safe and comfortable work environment for employees. All visitors sign in and are escorted by laboratory personnel. Access is controlled by various measures.

The laboratory is equipped with structural safety features. Each employee is familiar with the location, use, and capabilities of general and specialized safety features associated with their workplace. The laboratory provides and requires the use of protective equipment including safety glasses, protective clothing, gloves, etc. OSHA and other regulatory agency guidelines regarding required amounts of bench and fume hood space, lighting, ventilation (temperature and humidity-controlled), access, and safety equipment are met or exceeded.

Traffic flow through sample preparation and analysis areas is minimized to reduce the likelihood of contamination. Adequate floor space and bench top area is provided to allow unencumbered sample preparation and analysis space. Sufficient space is also provided for storage of reagents and media, glassware, and portable equipment. Ample space is also provided for refrigerated sample storage before analysis and archival storage of samples after analysis. Laboratory HVAC and deionized water systems are designed to minimize potential trace contaminants.

The laboratory is separated into specific areas for sample receiving, sample preparation, volatile organic sample analysis, non-volatile organic sample analysis, inorganic sample analysis, and administrative functions.

18.2 ENVIRONMENT

Laboratory accommodation, test areas, energy sources, lighting are adequate to facilitate proper performance of tests. The facility is equipped with heating, ventilation, and air conditioning (HVAC) systems appropriate to the needs of environmental testing performed at this laboratory.

The environment in which these activities are undertaken does not invalidate the results or adversely affect the required accuracy of any measurements.

The laboratory provides for the effective monitoring, control and recording of environmental conditions that may affect the results of environmental tests as required by the relevant specifications, methods, and procedures. Such environmental conditions include humidity, voltage, temperature, and vibration levels in the laboratory. A 225KVA UPS is installed in the main electrical bus to provide at least 15 minutes of backup power in the event of a power failure. This unit also provides voltage and frequency control of lab and office power. A spike/surge arrestor is installed to protect against power surge/sag and lightning strikes. A 30 KW natural gas-fueled backup generator is installed to provide power to the I.T. area in the event of a power failure. Additionally, this generator provides power to two walk-in sample

storage coolers and several other smaller sample storage coolers. Smaller portable generators are available to provide "spot power" where needed in the event of a power failure.

When any of the method or regulatory required environmental conditions change to a point where they may adversely affect test results, analytical testing must be discontinued until the environmental conditions are returned to the required levels.

Environmental conditions of the facility housing the computer network and LIMS are regulated to protect against raw data loss.

18.3 WORK AREAS

There is effective separation between neighboring areas when the activities therein are incompatible with each other. Examples include:

- Volatile organic chemical handling areas, including sample preparation and waste disposal, and volatile organic chemical analysis areas.

Access to, and use of, all areas affecting the quality of analytical testing is defined and controlled by secure access to the laboratory building as described below in the Building Security section.

Adequate measures are taken to ensure good housekeeping in the laboratory and to ensure that any contamination does not adversely affect data quality. These measures include regular cleaning to control dirt and dust within the laboratory.

Work areas are available to ensure an unencumbered work area. Work areas include:

- Access and entryways to the laboratory
- Sample receipt areas
- Sample storage areas
- Chemical and waste storage areas
- Data handling and storage areas
- Sample processing areas
- Sample analysis areas

18.4 FLOOR PLAN

A floor plan can be found in Appendix 1.

18.5 BUILDING SECURITY

Building keys are distributed to employees as necessary.

Visitors to the laboratory sign in and out in a visitor's logbook. A visitor is defined as any person who visits the laboratory who is not an employee of the laboratory. In addition to signing into

the laboratory, the Environmental, Health and Safety Manual contains requirements for visitors and vendors. There are specific safety forms that must be reviewed and signed.

Visitors (with the exception of company employees) are escorted by laboratory personnel at all times, or the location of the visitor is noted in the visitor's logbook.

Signs are posted in the laboratory designating employee only areas - "Authorized employees beyond this point".

SECTION 19

TEST METHODS AND METHOD VALIDATION
(NELAC 5.5.4)

19.1 OVERVIEW

The laboratory uses methods that are appropriate to meet our clients' requirements and that are within the scope of the laboratory's capabilities. These include sampling, handling, transport, storage and preparation of samples; and where appropriate, an estimation of the measurement of uncertainty as well as statistical techniques for analysis of environmental data.

Instructions are available in the laboratory for the operation of equipment as well as for the handling and preparation of samples. All instructions, Standard Operating Procedures (SOPs), reference methods and manuals relevant to the working of the laboratory are readily available to all staff. Deviations from published methods are documented (with justification) in the laboratory's approved SOPs. SOPs are submitted to clients for review at their request. Significant deviations from published methods require client approval and regulatory approval where applicable.

19.2 STANDARD OPERATING PROCEDURES (SOPs)

The laboratory maintains SOPs that accurately reflect all phases of the laboratory such as assessing data integrity, corrective actions, handling customer complaints as well as all analytical methods and sampling procedures. The method SOPs are derived from the most recently promulgated/approved, published methods and are specifically adapted to the laboratory facility. Modifications or clarifications to published methods are clearly noted in the SOPs. All SOPs are controlled in the laboratory.

- All SOPs contain a revision number, effective date, and appropriate approval signatures. Controlled copies are available to all staff.
- Procedures for writing an SOP are incorporated by reference to TestAmerica's Corporate SOP CW-Q-S-002 entitled Writing a Standard Operating Procedure, or the laboratory's SOP NC-QA-027, Preparation and Management of Standard Operating Procedures.
- SOPs are reviewed at a minimum of every two years (annually for Drinking Water and DoD SOPs), and where necessary, revised to ensure continuing suitability and compliance with applicable requirements.

19.3 LABORATORY METHODS MANUAL

For each test method, the laboratory must have available the published referenced method as well as the laboratory developed SOP.

Note: If more stringent standards or requirements are included in a mandated test method or regulation than those specified in this manual, the laboratory must demonstrate that such requirements are met. If it is not clear which requirements are more stringent, the standard from the method or regulation is to be followed. Any exceptions or deviations from the referenced methods or regulations are noted in the specific analytical SOP.

The laboratory maintains an SOP Index for both technical and non-technical SOPs. Technical SOPs are maintained to describe a specific test method. Non-technical SOPs are maintained to describe functions and processes not related to a specific test method.

19.4 SELECTION OF METHODS

Since numerous methods and analytical techniques are available, continued communication between the client and laboratory is imperative to assure the correct methods are utilized. Once client methodology requirements are established, this and other pertinent information is summarized by the Project Manager. These mechanisms ensure that the proper analytical methods are applied when the samples arrive for log-in. For non-routine analytical services, e.g., special matrices, non-routine compound lists, etc., the method of choice is selected based on client needs and available technology. The methods selected should be capable of measuring the specific parameter of interest, in the concentration range of interest, and with the required precision and accuracy.

19.4.1 Sources of Methods

Routine analytical services are performed using standard EPA-approved methodology. In some cases, modification of standard approved methods may be necessary to provide accurate analyses of particularly complex matrices. When the use of specific methods for sample analysis is mandated through project or regulatory requirements, only those methods must be used.

When clients do not specify the method to be used or methods are not required, the methods used must be clearly validated and documented in an SOP and available to clients and/or the end user of the data.

The analytical methods used by the laboratory are those currently accepted and approved by the U. S. EPA and the state or territory from which the samples were collected. Reference methods include:

- *Method 1664, Revision A: N-Hexane Extractable Material (HEM; Oil and Grease) and Silica Gel Treated N-Hexane Extractable Material (SGT-HEM); Non-polar Material) by Extraction and Gravimetry, EPA-821-R-98-002, February 1999*
- *Guidelines Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act, and Appendix A-C; 40 CFR Part 136, USEPA Office of Water. Revised as of July 1, 1995, Appendix A to Part 136 - Methods for Organic Chemical Analysis of Municipal and Industrial Wastewater (EPA 600 Series)*
- *Methods for Chemical Analysis of Water and Wastes, EPA 600 (4-79-020), 1983.*
- *Methods for the Determination of Inorganic Substances in Environmental Samples, EPA-600/R-93/100, August 1993.*
- *Methods for the Determination of Metals in Environmental Samples, EPA/600/4-91/010, June 1991. Supplement I: EPA-600/R-94/111, May 1994.*
- *Standard Methods for the Examination of Water and Wastewater, 18th/19th /20th edition/ on-line edition Eaton, A.D. Clesceri, L.S. Greenberg, A.E. Eds; American Water Works Association, Water Pollution Control Federation, American Public Health Association: Washington, D.C.*

- *Test Methods for Evaluating Solid Waste Physical/Chemical Methods (SW846), Third Edition, September 1986, Final Update I, July 1992, Final Update IIA, August 1993, Final Update II, September 1994; Final Update IIB, January 1995; Final Update III, December 1996.*
- *Annual Book of ASTM Standards, American Society for Testing & Materials (ASTM), Philadelphia, PA.*
- *Code of Federal Regulations (CFR) 40, Parts 136, 141, 172, 173, 178, 179 and 261*

TABLE 19-1. Wet Chemistry Methods [1]

Analytical Parameters	Matrix	Fields of Testing		
		CWA	RCRA (SW846)	Other
Acidity	Water	305. [2] SM 2310 B	--	--
Alkalinity, Bicarbonate, Carbonate	Water	305. [2] SM 2320 B		
	Solid	EPA 310.1[2] (M)	--	--
Arsenic (ASV) Anodic Stripping Voltammetry	Water	--	EPA 7063	--
Ash Content	Solid	--	--	ASTM D29-74
Biochemical Oxygen Demand, Carbonaceous	Water	EPA 405.1 SM 5210 B	--	--
Bromide	Water	EPA 300.0A	EPA 9056A	--
	Waste	EPA 300.0A	EPA 9056A	--
	Solid	EPA 300.0A (M)	EPA 9056A	--
Cation-Exchange Capacity	Solid	--	EPA 9081	--
Chemical Oxygen Demand	Water	EPA 410.4 SM 5220D	--	--
	Waste	EPA 410.4	--	--
Chloride	Water	EPA 300.0A EPA 325.2[2]	EPA 9056A EPA 9251	EPA 325.2[2]
	Waste	EPA 300.0A	EPA 9056A	--
	Solid	EPA 300.0A (M)	EPA 9056A EPA 9251(M)	--
Chromium, Hexavalent	Water	EPA 3500-Cr-D	EPA 7196A	--
	Waste	EPA 3500-Cr-D	EPA 7196A	--

Analytical Parameters	Matrix	Fields of Testing		
		CWA	RCRA (SW846)	Other
	Solid	--	EPA 3060A EPA 7196A	--
Specific Conductance	Water	EPA 120.1 SM 2510B	EPA 9050A	--
	Waste	EPA 120.1	EPA 9050A	--
	Solid	--	EPA 9050A	--
Chlorine, Residual	Water	EPA 330.5[2] SM 3500 CL-G	--	--
Cyanide (Amenable)	Water	EPA 335.1[2] SM 4500 CN-G	EPA 9012A	--
	Solid	--	EPA 9012A	--
Cyanide (Total)	Water	SM 4500-CN E EPA 335.4 335.2-CLP-M	EPA 9012A	---
	Waste	--	EPA 9012A	--
	Solid	335.2-CLP-M	EPA 9012A	--
Cyanide (Weak and Dissociable) (Free)	Water	SM 4500-CN I	--	--
Dissolved Oxygen	Water	360.1[2] SM 4500 O-G	--	--
Flash Point	Waste	--	EPA 1010	ASTM D93-9
	Solid	--	EPA 1010	ASTM D93-9
Fluoride	Water	EPA 300.0A EPA 340.2[2]	EPA 9056A	SM 4500 F-C, ISE
	Waste	EPA 340.2 (M)[2] EPA 300.0A (M)	EPA 9056A	--
	Solid	EPA 340.2 (M)[2] EPA 300.0A (M)	EPA 9056A	--
Iron, Ferrous & Ferric	Water	SM 3500 FE D	--	--
Hardness	Water	EPA 130.2[2]	--	SM 2340B
Moisture	Solid	---	EPA 160.3 (M) ASTM D2216-90	---
Nitrogen, Ammonia	Water	EPA 350.1	--	EPA 350.2[2]
	Waste	EPA 350.1	--	EPA 350.2[2]
	Solid	EPA 350.1	--	EPA 350.2[2]
	Water	SM 4500 NH_3-E (Titration)	--	--

Analytical Parameters Matrix	Matrix	Fields of Testing		
		CWA	RCRA (SW846)	Other
Nitrogen, Ammonia (cont'd)	Water	SM 4500 NH_3-F (ISE)	--	--
Nitrite (NO_2)	Water	EPA 300.0A	EPA 9056A	--
	Waste	EPA 300.0A (M)	EPA 9056A	--
	Solid	EPA 300.0A (M)	EPA 9056A	--
Nitrate (NO_3)	Water	EPA 300.0A	EPA 9056A	SM 4500 NO_3-E
	Waste	EPA 300.0A (M)	EPA 9056A	--
	Solid	EPA 300.0A (M)	--	--
Nitrate plus Nitrite NO_2/NO_3	Water	EPA 353.2	--	--
	Waste	EPA 353.2	--	--
Total Kjeldahl Nitrogen (TKN)	Water	EPA 351.3	--	SM 4500 NO_3
	Waste	EPA 351.3	--	--
	Solid	EPA 351.3	--	--
Oil and Grease (Hexane Extractable Material)	Water	EPA 1664A	EPA 9071B	--
	Waste	EPA 1664A	EPA 9071B	--
	Solid	--	EPA 9071B	--
Ortho-phosphate o-PO_4	Water	EPA 300.0A EPA 365.1	EPA 9056A	SM 4500 P-E
	Waste	EPA 300.0A (M)	EPA 9056A	--
	Solid	EPA 300.0A (M) EPA 365.1	EPA 9056A	--
pH	Water	EPA 150.1[2]	EPA 9040B	EPA 9041
	Waste	SM 4500 H-B	EPA 9045C	--
	Solid	---	EPA 9045C	--
Paint Filter	Water	--	EPA 9095A	--
Phenolics	Water	EPA 420.1	--	--
	Waste	--	EPA 9065	--
	Solid	--	EPA 9065	--
Phosphorus (Total)	Water	EPA 365.1	--	SM 4500 P-E
	Waste	EPA 365.1	--	--
	Solid	EPA 365.1	--	--
Sulfate (SO_4)	Water	EPA 300.0A EPA 375.4[2]	EPA 9056A EPA 9038	--
	Waste	EPA 300.0A (M) EPA 375.4[2]	EPA 9056A EPA 9038	--
	Solid	EPA 300.0A (M)	EPA 9056A EPA 9038 (M)	--

Analytical Parameters	Matrix	Fields of Testing		
		CWA	RCRA	Other
Sulfide	Water	EPA 376.1[2]	EPA 9030A SM 4500	9030B/9034
Total Organic Carbon (TOC)	Water	EPA 415.1[2]	EPA 9060	SM 5310 D
	Waste	--	EPA 9060	--
	Solid	EPA 415.1 (M)	EPA 9060 (M)	Walkley-Black
Total Organic Halides (TOX)	Water	--	EPA 9020B EPA 9023(EOX)	EPA 450.1
	Waste	--	--	--
	Solid	--	EPA 9020B	--
Total Petroleum Hydrocarbons	Water	EPA 1664A (SGT-HEM)	EPA 9071B	--
	Waste	EPA 1664A (SGT-HEM)	EPA 9071B	--
	Solid	--	EPA 9071B	--
Total Solids	Water	EPA 160.3	--	--
	Waste	EPA 160.3	--	--
	Solid	EPA 160.3 (M)	--	--
Total Dissolved Solids	Water	EPA 160.1	--	2540E
Total Suspended Solids	Water	EPA 160.2	---	2540E
Volatile and Volatile Suspended Solids	Water	EPA 160.4	--	--
Settleable Solids	Water	EPA 160.5	--	--
Turbidity	Water	EPA 180.1	--	--

[1] Any matrix not listed is not applicable for the associated method
[2] Removed from 40CFR

TABLE 19-2. Methods for Mercury by Cold Vapor Atomic Absorption

Analytical Parameters	Matrix	Fields of Testing			
			CWA	RCRA (SW846)	Other
Mercury (CVAA)	Water		EPA 245.1	EPA 7470A	--
	TCLP Leachate		--	EPA 7470A	--
	Waste		--	EPA 7471A	--
	Solid		EPA 254.5	EPA 7471A	--

TABLE 19-3. Methods for Mercury by Cold Vapor Atomic Fluororescence

Analytical Parameters	Matrix	Fields of Testing			
			CWA	RCRA (SW846)	Other
Mercury, Low Level (CVAFS)	Water		--	--	EPA 1631E

TABLE 19-4. Methods for Metals by ICP and ICPMS

Analytical Parameters	Matrix	Fields of Testing			
			CWA	RCRA (SW846)	Other
Aluminum	Water		EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
	Waste		---	EPA 6010B EPA 6020	---
	Solid		EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
Antimony	Water		EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
	Waste		---	EPA 6010B EPA 6020	---
	Solid		EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
Arsenic	Water		EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
	Waste		---	EPA 6010B EPA 6020	---
	Solid		EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---

Analytical Parameters	Matrix	Fields of Testing			
			CWA	RCRA (SW846)	Other
Barium	Water		EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
	Waste		---	EPA 6010B EPA 6020	---
	Solid		EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
Beryllium	Water		EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
	Waste		---	EPA 6010B EPA 6020	---
	Solid		EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
Boron	Water		EPA 200.7	EPA 6010B	---
	Waste		---	EPA 6010B	---
	Solid		EPA 200.7	EPA 6010B	---
Calcium	Water		EPA 200.7	EPA 6010B	---
	Waste		---	EPA 6010B	---
	Solid		EPA 200.7	EPA 6010B	---
Cadmium	Water		EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
	Waste		---	EPA 6010B EPA 6020	---
	Solid		EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
Cobalt	Water		EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
	Waste		---	EPA 6010B EPA 6020	---
	Solid		EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
Chromium	Water		EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
	Waste		---	EPA 6010B EPA 6020	---
	Solid		EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
Copper	Water		EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
	Waste		---	EPA 6010B EPA 6020	---
	Solid		EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---

Analytical Parameters	Matrix	Fields of Testing			
			CWA	RCRA (SW846)	Other
Iron	Water		EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	
	Waste		---	EPA 6010B EPA 6020	---
	Solid		EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
Lead	Water		EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
	Waste		---	EPA 6010B EPA 6020	---
	Solid		EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
Magnesium	Water		EPA 200.7	EPA 6010B EPA 6020	---
	Waste		---	EPA 6010B EPA 6020	---
	Solid		EPA 200.7	EPA 6010B EPA 6020	---
Manganese	Water		EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
	Waste		---	EPA 6010B EPA 6020	---
	Solid		EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
Molybdenum	Water		EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
	Waste		---	EPA 6010B EPA 6020	---
	Solid		EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
Nickel	Water		EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
	Waste		---	EPA 6010B EPA 6020	---
	Solid		EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
Potassium	Water		EPA 200.7	EPA 6010B	---
	Waste		---	EPA 6010B	---
	Solid		---	EPA 6010B	---
Selenium	Water		EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
	Waste		---	EPA 6010B EPA 6020	---
	Solid		EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---

Analytical Parameters	Matrix	Fields of Testing			
			CWA	RCRA (SW846)	Other
Silver	Water		EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
	Waste		---	EPA 6010B EPA 6020	---
	Solid		EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
Sodium	Water		EPA 200.7	EPA 6010B	---
	Waste		---	EPA 6010B	---
	Solid		---	EPA 6010B	---
Tin	Water		EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
	Waste		---	EPA 6010B EPA 6020	---
	Solid		EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
Thallium	Water		EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
	Waste		---	EPA 6010B EPA 6020	---
	Solid		EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
Titanium	Water		EPA 200.7	EPA 6010B	---
	Waste		---	EPA 6010B	---
	Solid		---	EPA 6010B	---
Vanadium	Water		EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
	Waste		---	EPA 6010B EPA 6020	---
	Solid		---	EPA 6010B EPA 6020	---
Zinc	Water		EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
	Waste		---	EPA 6010B EPA 6020	---
	Solid		EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---

TABLE 19-5. Metals Sample Preparation Methods

Analytical Parameters	Matrix		Fields of Testing		
			CWA	RCRA (SW846)	Other
Toxicity Characteristic Leaching Procedure (TCLP)	Water		---	EPA 1311	---
	Waste		---	EPA 1311	---
	Solid		---	EPA 1311	---
ICP Metals	Water		EPA 200.7	EPA 3005A EPA 3010A	---
	TCLP Leachate		---	EPA 3010A	---
	Waste		---	EPA 3050B	---
	Solid		---	EPA 3050B	---
ICPMS Metals	Water		EPA 200.8	EPA 3010A	---
	TCLP		---	EPA 3010A	---
	Waste		---	EPA 3050B	---
	Solid		---	EPA 3050B	---
CVAA Mercury	Water		EPA 245.1	EPA 7470A	---
	TCLP Leachate		---	EPA 7470A	---
	Waste		---	EPA 7471A	---
	Solid		---	EPA 7471A	
CVAFS Mercury Low Level	Water		---	---	EPA 1631E

TABLE 19-6. Organic Sample Preparation Methods

Analytical Parameters	Matrix	Fields of Testing		
		CWA	RCRA (SW846)	Other
Volatiles by GC/MS	Water	EPA 624	EPA 5030B	---
	Waste	---	EPA 5030B EPA 5035	---
	Solid	---	EPA 5035 EPA 5035A	---
Halogenated Volatiles by GC	Water	EPA 601	EPA 5030B	---
	Waste	---	EPA 5030B EPA 5035	---
	Solid	---	EPA 5035 EPA 5035A	---
Aromatic Volatiles by GC	Water	EPA 602	EPA 5030B	---
	Waste	---	EPA 5030B EPA 5035	---
	Solid	---	EPA 5035 EPA 5035A	---
Semivolatiles by GC/MS	Water	EPA 625	EPA 3510C EPA 3520C	---
	TCLP Leachate	---	EPA 3510C EPA 3520C	---
	Waste	---	EPA 3550B EPA 3540C EPA 3580A EPA 3541	---
	Solid	---	EPA 3550B EPA 3540C EPA 3541	---
Pesticides/PCBs by GC	Water	EPA 608	EPA 3510C EPA 3520C	---
	TCLP Leachate	---	EPA 3510C EPA 3520C	---
	Waste	---	EPA 3550B EPA 3540C EPA 3580A EPA 3541	---
	Solid	---	EPA 3550B EPA 3540C EPA 3541	---

Analytical Parameters	Matrix		Fields of Testing		
			CWA	RCRA (SW846)	Other
Herbicides by GC	Water		EPA 615	EPA 8151A	---
	Waste		---	EPA 8151A	---
	Solid		---	EPA 8151A	---
Total Petroleum Hydrocarbons (Gasoline Range) by GC	Water		---	EPA 5030B	WI GRO
	Waste		---	EPA 5030B EPA 5035	WI GRO
	Solid		---	EPA 5035 EPA 5035	WI GRO
Total Petroleum Hydrocarbons (Diesel Range) by GC	Water		---	EPA 3510C EPA 3520C	WI DRO
	TCLP Leachate		---	EPA 3510C EPA 3520C	---
	Waste		---	EPA 3550B EPA 3580A	WI DRO
	Solid		---	EPA 3550B	WI DRO

TABLE 19-7. Organic Methods of Analysis

Analytical Parameters	Matrix		Fields of Testing		
			CWA	RCRA (SW846)	Other
Volatiles by GC/MS	Water		EPA 624	EPA 8260B	---
	Waste		---	EPA 8260B	---
	Solid		---	EPA 8260B	---
Halogenated Volatiles by GC	Water		EPA 601	EPA 8021B	---
	Waste		---	EPA 8021B	---
	Solid		---	EPA 8021B	---
Aromatic Volatiles by GC	Water		EPA 602	EPA 8021B	---
	Waste		---	EPA 8021B	---
	Solid		---	EPA 8021B	---
Semivolatiles by GC/MS	Water		EPA 625	EPA 8270C	
	Waste		---	EPA 8270C	---
	Solid		---	EPA 8270C	---
Pesticides/PCBs by GC	Water		EPA 608	Pesticides 8081A PCBs 8082	---
	TCLP Leachate		---	Pesticides 8081A PCBs 8082	---
	Waste		---	Pesticides 8081A PCBs 8082	---
	Solid		---	Pesticides 8081A PCBs 8082	---
Phenoxyacid Herbicides by GC	Water		---	EPA 8151A	---
	TCLP Leachate		---	EPA 8151A	---
	Waste		---	EPA 8151A	---
	Solid		---	EPA 8151A	---
Gasoline Range Organics by GC	Water		---	EPA 8015B (M)	WI GRO
	Waste		---	EPA 8015B (M)	---
	Solid		---	EPA 8015B (M)	WI GRO
Total Petroleum Hydrocarbons (Diesel Range) by GC/FID	Water		---	EPA 8015B (M)	WI DRO
	Waste		---	EPA 8015B (M)	---
Dissolved Gases RSK-175	Water		---	---	SOP
Formaldehyde Carbonyl Compounds	Water		---	EPA 8315	---

The laboratory reviews updated versions to all the aforementioned references for adaptation based upon capabilities, instrumentation, etc., and implements them as appropriate. As such, the laboratory strives to perform only the latest versions of each approved method as regulations allow or require.

Other reference procedures for non-routine analyses may include methods established by specific states (e.g., Underground Storage Tank methods), ASTM or equipment manufacturers. Sample type, source, and the governing regulatory agency requiring the analysis will determine the method utilized.

The laboratory must inform the client when a method proposed by the client may be inappropriate or out of date. After the client has been informed, and they wish to proceed contrary to the laboratory's recommendation, it must be documented.

19.4.2 Demonstration of Capability

Before the laboratory may institute a new method and begin reporting results, the laboratory must confirm that it can properly operate the method. In general, this demonstration does not test the performance of the method in real world samples, but in an applicable and available clean matrix sample. If the method is for the testing of analytes that are not conducive to spiking, demonstration of capability may be performed on quality control samples.

19.4.2.1 A demonstration of capability is performed (SOP NC-QA-028, Employee Orientation and Training) whenever there is a change in instrument type (e.g., new instrumentation), method, or personnel.

19.4.2.2 The initial demonstration of capability must be thoroughly documented and approved by the Technical Director and QA Manager prior to independently analyzing client samples. All associated documentation must be retained in accordance with the laboratories archiving procedures.

19.4.2.3 The laboratory must have an approved SOP, demonstrate satisfactory performance, and conduct an MDL study (when applicable). There may be other requirements as stated within the published method or regulations (i.e., retention time window study).

Note: In some instances, a situation may arise where a client requests that an unusual analyte be reported using a method where this analyte is not normally reported. If the analyte is being reported for regulatory purposes, the method must meet all procedures outlined within this QA Manual (SOP, MDL, and Demonstration of Capability). If the client states that the information is not for regulatory purposes, the result may be reported as long as the following criteria are met:

- The instrument is calibrated for the analyte to be reported using the criteria for the method and ICV/CCV criteria are met (unless an ICV/CCV is not required by the method or criteria are per project DQOs).
- The laboratory's nominal or default reporting limit (RL) is equal to the quantitation limit (QL), must be at or above the lowest non-zero standard in the calibration curve (low standard at or below the QL)and must be reliably determined. Project RLs are client specified reporting levels which may be higher than the QL. Results reported

below the QL must be qualified as estimated values. Also see Section 19.6.1.3, Relationship of Limit of Detection (LOD) to Quantitation Limit (QL).

Note: For Ohio VAP work, the term Reporting Limit will be used.

- The client request is documented and the lab informs the client of its procedure for working with unusual compounds. The final report must be footnoted as "*Reporting Limit based on the low standard of the calibration curve".*

19.4.3 Initial Demonstration of Capability (IDOC) Procedures

19.4.3.1 At least four aliquots must be prepared (including any applicable clean-up procedures) and analyzed according to the test method (either concurrently or over a period of days).

19.4.3.2 Using all of the results, calculate the mean recovery in the appropriate reporting units and the standard deviations for each parameter of interest. Refer to SOP NC-QA-028, Employee Orientation and Training, for details on this procedure.

Note: Results of successive LCS analyses can be used to fulfill the DOC requirement.

19.4.3.3 A certification statement (see Figure 19-1 as an example) must be used to document the completion of each initial demonstration of capability. A copy of the certification is archived in the analyst's training folder.

19.5 LABORATORY-DEVELOPED METHODS AND NON-STANDARD METHODS

Any new method developed by the laboratory must be fully defined in an SOP and validated by qualified personnel with adequate resources to perform the method. Method specifications and the relation to client requirements must be clearly conveyed to the client if the method is a non-standard method (not a published or routinely accepted method). The client must also be in agreement to the use of the non-standard method.

19.6 VALIDATION OF METHODS

Validation is the confirmation by examination and the provision of objective evidence that the particular requirements for a specific intended use are fulfilled.

All non-standard methods, laboratory designed/developed methods, standard methods used outside of their scope, and major modifications to published methods must be validated to confirm they are fit for their intended use. The validation will be as extensive as necessary to meet the needs of the given application. The results are documented with the validation procedure used and contain a statement as to the fitness for use.

19.6.1 Method Validation and Verification Activities for All New Methods

While method validation can take various courses, the following activities can be required as part of method validation. Method validation records are designated QC records and are archived accordingly.

19.6.1.1 Determination of Method Selectivity

Method selectivity is the demonstrated ability to discriminate the analyte(s) of interest from other compounds in the specific matrix or matrices from other analytes or interference. In some cases to achieve the required selectivity for an analyte, a confirmation analysis is required as part of the method.

19.6.1.2 Determination of Method Sensitivity

Sensitivity can be both estimated and demonstrated. Whether a study is required to estimate sensitivity depends on the level of method development required when applying a particular measurement system to a specific set of samples. Where estimations and/or demonstrations of sensitivity are required by regulation or client agreement, such as the procedure in 40 CFR Part 136 Appendix B, under the Clean Water Act, these shall be followed.

19.6.1.3 Relationship of Limit of Detection (LOD) to the Quantitation Limit (QL)

An important characteristic of expression of sensitivity is the difference in the LOD and the QL. The LOD is the minimum level at which the presence of an analyte can be reliably concluded. The QL is the minimum concentration of analyte that can be quantitatively determined with acceptable precision and bias. For most instrumental measurement systems, there is a region where semi-quantitative data is generated around the LOD (both above and below the estimated MDL or LOD) and below the QL. In this region, detection of an analyte may be confirmed but quantification of the analyte is unreliable within the accuracy and precision guidelines of the measurement system. When an analyte is detected below the QL, and the presence of the analyte is confirmed by meeting the qualitative identification criteria for the analyte, the analyte can be reliably reported, but the amount of the analyte can only be estimated. If data is to be reported in this region, it must be done so with a qualification that denotes the semi-quantitative nature of the result.

19.6.1.4 Determination of Interferences

A determination that the method is free from interferences in a blank matrix is performed.

19.6.1.5 Determination of Range

Where appropriate to the method, the quantitation range is determined by comparison of the response of an analyte in a curve to established or targeted criteria. Generally the upper quantitation limit is defined by highest acceptable calibration concentration. The lower quantitation limit or QL cannot be lower than the lowest non-zero calibration level, and can be constrained by required levels of bias and precision.

19.6.1.6 Determination of Accuracy and Precision

Accuracy and precision studies are generally performed using replicate analyses, with a resulting percent recovery and measure of reproducibility (standard deviation, relative standard deviation) calculated and measured against a set of target criteria.

19.6.1.7 Documentation of Method

The method is formally documented in an SOP. If the method is a minor modification of a standard laboratory method that is already documented in an SOP, an SOP Attachment describing the specific differences in the new method is acceptable in place of a separate SOP.

19.6.1.8 Continued Demonstration of Method Performance

Continued demonstration of Method Performance is addressed in the SOP. Continued demonstration of method performance is generally accomplished by batch-specific QC samples such as LCS, method blanks, or PT samples.

19.7 METHOD DETECTION LIMITS (MDL)/ LIMITS OF DETECTION (LOD)

Method detection limits (MDL) are initially determined in accordance with 40 CFR Part 136, Appendix B, or alternatively by other technically acceptable practices that have been accepted by regulators. MDL is also sometimes referred to as Limit of Detection (LOD). The MDL theoretically represents the concentration level for each analyte within a method at which the Analyst is 99% confident that the true value is not zero. The MDL is determined for each analyte initially during the method validation process and updated as required in the analytical methods, whenever there is a significant change in the procedure or equipment, or based on project specific requirements (refer to Section 19.7.10). Generally, the analyst prepares at least seven replicates of solution spiked at one to five times the estimated method detection limit (most often at the lowest standard in the calibration curve) into the applicable matrix with all the analytes of interest. Each of these aliquots is extracted (including any applicable clean-up procedures) and analyzed in the same manner as the samples. Where possible, the seven replicates should be analyzed over 2-4 days to provide a more realistic MDL. To allow for some flexibility, this low level standard may be analyzed every batch or every week or some other frequency rather than doing the study all at once. In addition, a larger number of data points may be used if the appropriate t-value multiplier is used.

Refer to the Corporate SOP CA-Q-S-006 or the laboratory's SOP NC-QA-021 for details on the laboratory MDL process.

Note: For Ohio VAP projects, the MDL procedure must also comply with OAC Rule 3745-300-01(A)(78).

19.8 INSTRUMENT DETECTION LIMITS (IDL)

19.8.1 The IDL is sometimes used to assess the reasonableness of the MDLs or in some cases required by the analytical method or program requirements. IDLs are most used in metals analyses but may be useful in demonstration of instrument performance in other areas.

19.8.2 IDLs are calculated to determine an instrument's sensitivity independent of any preparation method. IDLs are calculated either by using seven replicate spike analyses, like MDL but without sample preparation, or by the analysis of ten instrument blanks and calculating three times the absolute value of the standard deviation.

19.8.3 If IDL is > than the MDL, it may be used as the reported MDL.

19.9 VERIFICATION OF DETECTION AND REPORTING LIMITS

19.9.1 Once the MDL is determined, it must be verified on each instrument used for the given method. TestAmerica defines the DoD QSM Detection Limit (DL) as being equal to the MDL. TestAmerica also defines the DoD QSM Limit of Detection (LOD) as being equal to the lowest concentration standard that successfully verifies the MDL, also referred to as the MDLV standard. MDL and MDLV standards are extracted/digested and analyzed through the entire analytical process. The MDL and MDLV determinations do not apply to methods that are not readily spiked (e.g. pH, turbidity, etc.) or where the lab does not report to the MDL. If the MDLV standard is not successful, then the laboratory will redevelop their MDL. Initial and quarterly verification is required for all methods listed in the laboratory's DoD ELAP Scope of Accreditation. Refer to the laboratory SOP NC-QA-021 or Corporate CA-Q-S-006 for further details.

19.9.2 The laboratory quantitation limit is equivalent to the DoD Limit of Quantitation (LOQ), which is at a concentration equal to or greater than the lowest non-zero calibration standard. The DoD QSM requires the laboratory to perform an initial characterization of the bias and precision at the LOQ and quarterly LOQ verifications thereafter. If the quarterly verification results are not consistent with three-standard deviation confidence limits established initially, then the bias and precision will be reevaluated and clients contacted for any on-going projects. For DoD projects, TestAmerica makes a distinction between the Reporting Limit (RL) and the LOQ. The RL is a level at or above the LOQ that is used for specific project reporting purposes, as agreed to between the laboratory and the client. The RL cannot be lower than the LOQ concentration, but may be higher.

19.10 RETENTION TIME WINDOWS

Most organic analyses and some inorganic analyses use chromatography techniques for qualitative and quantitative determinations. For every chromatography analysis each analyte will have a specific time of elution from the column to the detector. This is known as the analyte's retention time. The variance in the expected time of elution is defined as the retention time window. As the key to analyte identification in chromatography, retention time windows must be established on every column for every analyte used for that method. These records are kept in each department. Complete details are available in the laboratory SOPs.

19.11 EVALUATION OF SELECTIVITY

The laboratory evaluates selectivity by following the checks within the applicable analytical methods, which include mass spectral tuning, second column confirmation, ICP interelement interference checks, chromatography retention time windows, sample blanks, atomic absorption, or fluorescence profiles.

19.12 ESTIMATION OF UNCERTAINTY OF MEASUREMENT

19.12.1 Uncertainty is "a parameter associated with the result of a measurement, that characterizes the dispersion of the values that could reasonably be attributed to the measurand" (as defined by the International Vocabulary of Basic and General Terms in Metrology, ISO Geneva, 1993, ISBN 92-67-10175-1). Knowledge of the uncertainty of a measurement provides additional confidence in a result's validity. Its value accounts for all the factors which could possibly affect the result, such as adequacy of analyte definition, sampling, matrix effects and interferences, climatic conditions, variances in weights, volumes, and standards, analytical procedure, and random variation. Some national accreditation organizations require the use of an "expanded uncertainty": the range within which the value of the measurand is believed to lie within at least a 95% confidence level with the coverage factor k=2.

19.12.2 Uncertainty is not error. Error is a single value, the difference between the true result and the measured result. On environmental samples, the true result is never known. The measurement is the sum of the unknown true value and the unknown error. Unknown error is a combination of systematic error, or bias, and random error. Bias varies predictably, constantly, and independently from the number of measurements. Random error is unpredictable, assumed to be Gaussian in distribution, and reducible by increasing the number of measurements.

19.12.3 The minimum uncertainty associated with results generated by the laboratory can be determined by using the Laboratory Control Sample (LCS) accuracy range for a given analyte. The LCS limits are used to assess the performance of the measurement system since they take into consideration all of the laboratory variables associated with a given test over time (except for variability associated with the sampling and the variability due to matrix effects). The percent recovery of the LCS is compared either to the method-required LCS accuracy limits or to the statistical, historical, in-house LCS accuracy limits.

19.12.4 To calculate the uncertainty for the specific result reported, multiply the result by the decimal of the lower end of the LCS range percent value for the lower end of the uncertainty range, and multiply the result by the decimal of the upper end of the LCS range percent value for the upper end of the uncertainty range. These calculated values represent a 99%-certain range for the reported result. As an example, suppose that the result reported is 1.0 mg/l, and the LCS percent recovery range is 50 to 150%. The uncertainty range would be 0.5 to 1.5 mg/l, which could also be written as 1.0 ± 0.5 mg/l.

19.12.5 In the case where a well recognized test method specifies limits to the values of major sources of uncertainty of measurement, e.g., 524.2, 525, etc., and specifies the form of presentation of calculated results, no further discussion of uncertainty is required.

19.13 CONTROL OF DATA

The laboratory has policies and procedures in place to ensure the authenticity, integrity, and accuracy of the analytical data generated by the laboratory.

19.13.1 Computer and Electronic Data Related Requirements

The three basic objectives of our computer security procedures and policies are shown below. The laboratory is currently running the QuantIMS LIMS which is a custom in-house developed LIMS system that has been highly customized to meet the needs of the laboratory. It is referred to as LIMS for the remainder of this section. The LIMS utilizes AS400 which is an industry standard relational database platform. It is referred to as Database for the remainder of this section.

19.13.1.1 Maintain the Database Integrity

Assurance that data is reliable and accurate through data verification (review) procedures, password-protecting access, anti-virus protection, data change requirements, as well as an internal LIMS permissions procedure.

- LIMS Database Integrity is achieved through data input validation, internal user controls, and data change requirements.

- Spreadsheets and other software developed in-house must be verified with documentation through hand calculations prior to use.

19.13.1.2 Ensure Information Availability

Protection against loss of information or service is ensured through scheduled back-ups, stable file server network architecture, secure storage of media, line filter, Uninterruptible Power Supply (UPS), and maintaining older versions of software as revisions are implemented.

19.13.1.3 Maintain Confidentiality

Ensure data confidentiality through physical access controls, and encryption of when electronically transmitting data.

19.13.2 Data Reduction

The complexity of the data reduction depends on the analytical method and the number of discrete operations involved, e.g., extractions, dilutions, instrument readings and concentrations. The analyst calculates the final results from the raw data or uses appropriate computer programs to assist in the calculation of final reportable values.

For manual data entry, e.g., Wet Chemistry, the data is reduced by the analyst and then verified by peer review once updated in LIMS. The review checklists are signed by both the analyst and reviewer to confirm the accuracy of the manual entry(s).

Manual integration of peaks will be documented and reviewed and the raw data will be flagged in accordance with the TestAmerica Corporate SOP CA-Q-S-002, Acceptable Manual Integration Practices.

Analytical results are reduced to appropriate concentration units specified by the analytical method, taking into account factors such as dilution, sample weight or volume, etc. Blank correction will be applied only when required by the method or per manufacturer's indication; otherwise, it must not be performed. Calculations are independently verified by appropriate laboratory staff. Calculations and data reduction steps for various methods are summarized in the respective analytical SOPs or program requirements.

19.13.2.1 All raw data must be retained. All criteria pertinent to the method must be recorded. The documentation is recorded at the time observations or calculations are made and must be signed or initialed/dated (month/day/year). It must be easily identifiable who performed which tasks if multiple people were involved.

19.13.2.2 In general, concentration results are reported in milligrams per liter (mg/l) or micrograms per liter (µg/l) for liquids and milligrams per kilogram (mg/kg) or micrograms per kilogram (µg/kg) for solids. The units "mg/l" and "mg/kg" are the same as "parts per million (ppm)". The units "µg/l" and "µg/kg" are the same as "parts per billion (ppb)." For values greater than 10,000 mg/l, results can be reported in percent, i.e., 10,000 mg/l = 1%. Units are defined in each lab SOP.

19.13.2.3 For those methods that do not have an instrument printout or an instrumental output compatible with the LIMS System, the raw results and dilution factors are entered directly into LIMS by the analyst, and the software calculates the final result for the analytical report. LIMS has a defined significant figure criterion for each analyte.

19.13.2.4 The laboratory strives to import data directly from instruments or calculation spreadsheets to ensure that the reported data are free from transcription and calculation errors. For those analyses with an instrumental output compatible with the LIMS, the raw results and dilution factors are transferred into LIMS electronically after reviewing the quantitation report, and removing unrequested or poor spectrally-matched compounds. The analyst prints a copy of what has been entered to check for errors. This printout and the instrument's printout of calibrations, concentrations, retention times, chromatograms, and mass spectra, if applicable, are retained with the data file.

19.13.3 Logbook / Worksheet Use Guidelines

Logbooks and worksheets are filled out in 'real time' and have enough information on them to trace the events of the applicable analysis/task (e.g., calibrations, standards, analyst, sample ID, date, time on short holding time tests, temperatures when applicable, calculations are traceable, etc.).

- Corrections are made following the procedures outlined in Section 12.
- Logbooks are controlled by the QA Department. A record is maintained of all logbooks in the lab.

- Unused portions of pages must be "Z"'d out, signed and dated.
- Worksheets are created with the approval of the QA Manager at the facility. The QA Manager controls all worksheets following the procedures in Section 6.

19.13.4 Review / Verification Procedures

19.13.4.1 Data Recording Procedures

To ensure data integrity, all documentation of data and records generated or used during the process of data generation must be performed in compliance with Section 3 of this document and Policy CA-Q-T-005, Laboratory Documentation.

19.13.4.2 Data Reduction and Verification Procedures

Data review procedures comprise a set of computerized and manual checks applied at appropriate levels of the measurement process. Data review begins with the reduction or processing of data and continues through verification of the data and the reporting of analytical results. Calculations are checked from the raw data to the final value prior to reporting results for each group of samples. Data reduction can be performed by the analyst who obtained the data or by another analyst. Data verification starts with the analyst who performs a 100% review of the data to ensure the work was done correctly the first time. Data verification continues with review by a second reviewer who verifies that data reduction has been correctly performed and that the analytical results correspond to the data acquired and processed.

19.13.4.2.1 Data Reduction and Initial Verification

Data reduction and initial verification may be performed by more than one analyst depending upon the analytical method employed. The preparation and analytical data may be reviewed independently by different analysts. In these instances, each item may not be applicable to the subset of the data verified or an item may be applicable in both instances. It is the responsibility of the analyst to ensure that the verification of data in his or her area is complete. The data reduction and initial verification process must ensure that:

- Sample preparation information is correct and complete including documentation of standard identification, solvent lot numbers, sample amounts, etc.
- Analysis information is correct and complete including proper identification of analysis output (charts, chromatograms, mass spectra, etc.)
- Analytical results are correct and complete including calculation or verification of instrument calibration, QC results, and qualitative and quantitative sample results with appropriate qualifiers
- The appropriate SOPs have been followed and are identified in the project and/or laboratory records
- Proper documentation procedures have been followed
- All non-conformances have been documented
- Special sample preparation and analytical requirements have been met.
- The data generated have been reported with the appropriate number of significant figures as defined by the analytical method in the LIMS or

otherwise specified by the client.

In general, data will be processed by an analyst in one of the following ways:

- Manual computation of results directly on the data sheet or on calculation pages attached to the data sheets
- Input of raw data for computer processing
- Direct acquisition and processing of raw data by a computer.

If data are manually processed by an analyst, all steps in the computation must be provided including equations used and the source of input parameters such as response factors (RFs), dilution factors, and calibration constants. If calculations are not performed directly on the data sheet, they may be attached to the data sheets.

Manual integrations are sometimes necessary to correct misintegrations by an automatic data system software program, but must only be performed when necessary. Further discussion of manual integrations and the required documentation is given in Policy CA-Q-S-002, Acceptable Manual Integration Practices.

For data that are input by an analyst and processed using a computer, a copy of the input must be kept and uniquely identified with the project number and other information as needed. The samples analyzed must be clearly identified.

If data are directly acquired from instrumentation or a test procedure and processed, or immediately entered into LIMS, the analyst must verify that the following are correct:

- Project and sample numbers
- Calibration constants and RFs
- Units
- Numerical values used for reporting limits.

Analysis-specific calculations for methods are provided in SOPs. In cases where computers perform the calculations, software must be validated or verified, as described in Section 6.0 of this document, before it is used to process data.

The data reduction is documented, signed and dated by the analyst completing the process. Initial verification of the data reduction by the same analyst is documented on a data review checklist, signed and dated by the analyst.

19.13.4.2.2 Data Verification

Following the completion of the initial verification by the analyst performing the data reduction, a systematic check of the data that has been fully reduced and checked through Level 1 review is performed by an experienced peer, group leader, or designee. This Level 2 check is performed to ensure that Level 1 review has been completed correctly and thoroughly. The second level

reviewer examines the data signed by the analyst. Any exceptions noted by the analyst must be reviewed. Included in this review is an assessment of the acceptability of the data with respect to:

- Adherence of the procedure used to the requested analytical method SOP
- Correct interpretation of chromatograms, mass spectra, etc.
- Correctness of numerical input when computer programs are used (checked randomly)
- Correct identification and quantitation of constituents with appropriate qualifiers
- Numerical correctness of calculations and formulas (checked randomly)
- Acceptability of QC data (100% review)
- Documentation that instruments were operating according to method specifications (calibrations, performance checks, etc.)
- Documentation of dilution factors, standard concentrations, etc.
- Sample holding time assessment.

This review also serves as verification that the process the analyst has followed is correct in regard to the following:

- The analytical procedure follows the methods and client-specific instructions.
- Nonconforming events have been addressed by corrective action as defined on a nonconformance memo
- Valid interpretations have been made during the examination of the data and the review comments of the initial reviewer are correct
- The package contains all of the necessary documentation for data review and report production and results are reported in a manner consistent with the method used for preparation of data reports.

The specific items covered in the second stage of data verification may vary according to the analytical method, but this review of the data must be documented by signing the same checklist.

19.13.4.2.3 Completeness Verification

A third-level review is performed by the reporting and project management staff. This review is required before results are submitted to clients. This review serves to verify the completeness of the data report and to ensure that project requirements are met for the analyses performed. The items to be reviewed are:

- Analysis results are present for every sample in the analytical batch, reporting group, or sample delivery group (SDG)

- Every parameter or target compound requested is reported with either a value or reporting limit
- All nonconformances, including holding time violations, and data evaluation statements that impact the data quality are accompanied by clearly expressed comments from the laboratory

- The final report contains all the supporting documentation required by the project, and is in either the standard TestAmerica format or in the client-required

format.

- Implement checks to monitor the quality of laboratory results using correlation of results for different parameters of a sample (for example, does the TOC results justify the concentration of organic compounds found by GC/MS.)

- A narrative to accompany the final report must be finalized by the PM. This narrative must include relevant comments collected during the earlier reviews.

The Quality Assurance Department performs data reviews per SOP CA-Q-S-004, Internal Auditing. For DoD work, 10% of all reports must undergo an internal data review.

19.13.5 Manual Integrations

Computerized data systems provide the analyst with the ability to re-integrate raw instrument data in order to optimize the interpretation of the data. Though manual integration of data is an invaluable tool for resolving variations in instrument performance and some sample matrix problems, when used improperly, this technique would make unacceptable data appear to meet quality control acceptance limits. Improper re-integrations lead to legally indefensible data, a poor reputation, or possible laboratory decertification. Because guidelines for re-integration of data are not provided in the methods and most methods were written prior to widespread implementation of computerized data systems, the laboratory trains all analytical staff on proper manual integration techniques using TestAmerica's Corporate SOP (CA-Q-S-002).

19.13.5.1 The analyst must adjust baseline or the area of a peak in some situations, for example when two compounds are not adequately resolved or when a peak shoulder needs to be separated from the peak of interest. The analyst must use professional judgment and common sense to determine when manual integrating is required. Analysts are encouraged to ask for assistance from a senior analyst or manager when in doubt.

19.13.5.2 Analysts must not increase or decrease peak areas to for the sole purpose of achieving acceptable QC recoveries that would have otherwise been unacceptable. The intentional recording or reporting of incorrect information (or the intentional omission of correct information) is against company principals and policy and is grounds for immediate termination.

19.13.5.3 Client samples, performance evaluation samples, and quality control samples are all treated equally when determining whether or not a peak area or baseline should be manually adjusted.

19.13.5.4 All manual integrations receive a second level review. Manual integrations must be indicated on an expanded scale "after" chromatograms such that the integration performed can be easily evaluated during data review. Expanded scale "before" chromatograms are also required for all manual integrations on QC parameters (calibrations, calibration verifications, laboratory control samples, internal standards, surrogates, etc.) unless the laboratory has another documented corporate-approved procedure in place that can demonstrate an active process for detection and deterrence of improper integration practices.

Figure 19-1. Example - Demonstration of Capability Documentation

Analyst Demonstration of Capability
Certification Statement

Analyst Name
Date:

Test Method:
SOP:
Matrix:

TestAmerica North Canton laboratory
4101 Shuffel Drive NW
North Canton, OH 44720
(330) 497-9396

We, the undersigned, CERTIFY that:

1. The analyst identified above, using the cited test method with the specifications in the cited SOP, which is in use at this facility for the analysis of samples under the TestAmerica Quality Assurance Plan, has met the Initial or Ongoing Demonstration of Capability.

2. The test method was performed by the analyst identified on this certification following the TestAmerica SOP.

3. A copy of the laboratory-specific SOP is available for all personnel on-site.

4. The data associated with the initial/ongoing demonstration of capability are true, accurate, complete and self-explanatory (*). These data are attached to this certification statement.

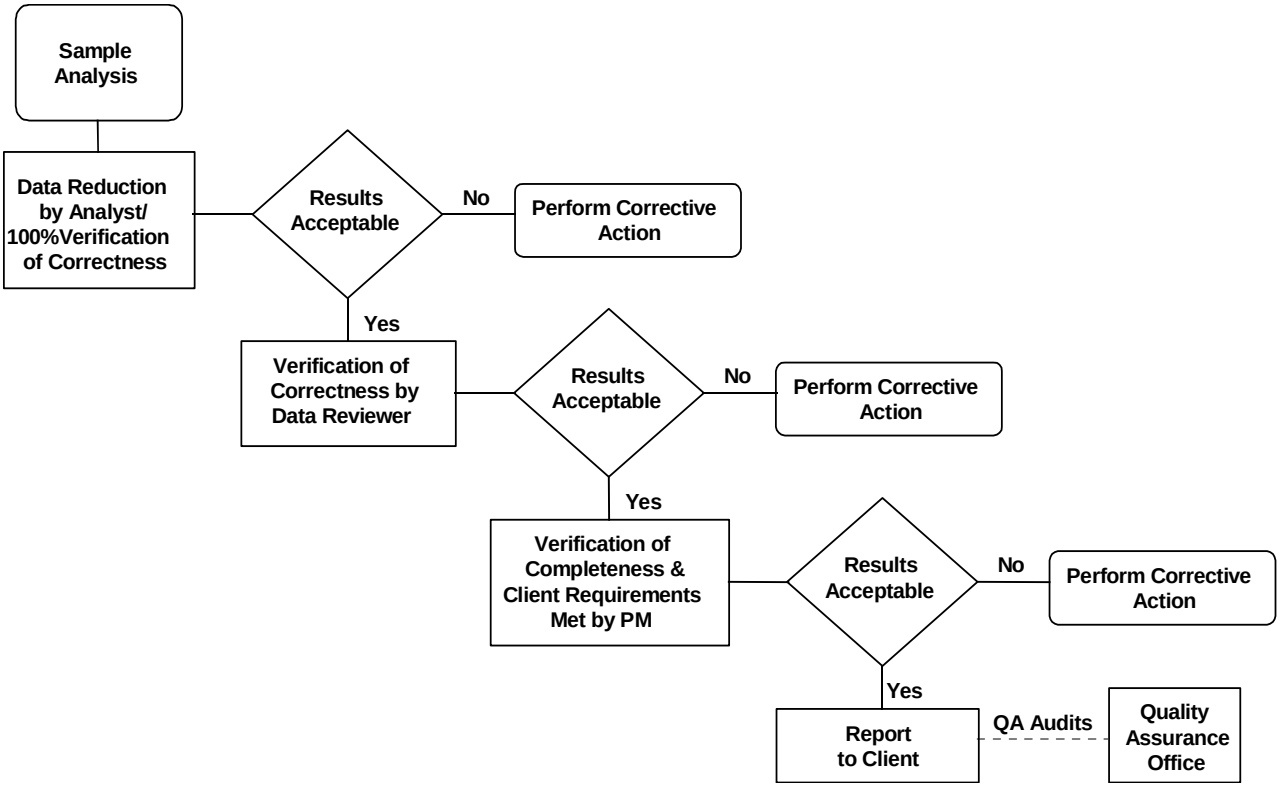
5. All raw data (including a copy of this certification form) necessary to reconstruct and validate these analyses have been retained at the facility, and that the associated information is well organized and available for review by authorized inspectors.

Comments/Observations:

Analyst's Name	Signature	Date
Technical Director's Name	Signature	Date
QA Manager's Name	Signature	Date

* *True: Consistent with supporting data.*
Accurate: Based on good laboratory practices consistent with sound scientific principles/practices.
Complete: Includes the results of all supporting performance testing.
Self-explanatory: Data properly labeled and stored so that the results are traceable and require no additional explanation.

Figure 19-2. Work Flow



SECTION 20

EQUIPMENT (AND CALIBRATIONS)
(NELAC 5.5.5)

20.1 OVERVIEW

The laboratory purchases the most technically advanced analytical instrumentation for sample analyses. Instrumentation is purchased on the basis of accuracy, dependability, efficiency and sensitivity. Each laboratory is furnished with all items of sampling, preparation, analytical testing and measurement equipment necessary to correctly perform the tests for which the laboratory has capabilities. Each piece of equipment is capable of achieving the required accuracy and complies with specifications relevant to the method being performed. Before being placed into use, the equipment (including sampling equipment) is calibrated and checked to establish that it meets its intended specification. The calibration routines for analytical instruments establish the range of quantitation. Calibration procedures are specified in laboratory SOPs. A list of laboratory equipment and instrumentation is presented in Table 20-1*.*

Equipment is only operated by authorized and trained personnel. Manufacturers instructions for equipment use are readily accessible to all appropriate laboratory personnel.

20.2 PREVENTIVE MAINTENANCE

20.2.1 The laboratory follows a well-defined maintenance program to ensure proper equipment operation and to prevent the failure of laboratory equipment or instrumentation during use. This program of preventive maintenance helps to avoid delays due to instrument failure.

20.2.2 Routine preventive maintenance procedures and frequency, such as lubrication, cleaning, and replacements, should be performed according to the procedures outlined in the manufacturer's manual. Qualified personnel must also perform maintenance when there is evidence of degradation of peak resolution, a shift in the calibration curve, loss of sensitivity, or failure to continually meet one of the quality control criteria.

20.2.3 Table 20-2 lists examples of scheduled routine maintenance. It is the responsibility of each Group Leader to ensure instrument maintenance logs are kept for all equipment in his/her department. Preventative maintenance procedures are also be outlined in analytical SOPs or instrument manuals. (Note: For some equipment, the log used to monitor performance is also the maintenance log. Multiple pieces of equipment may share the same log as long as it is clear as to which instrument is associated with an entry.)

20.2.4 Instrument maintenance logs are controlled and are used to document instrument problems, instrument repair and maintenance activities. Maintenance logs must be kept for all major pieces of equipment. Instrument Maintenance Logbooks may also be used to specify instrument parameters.

20.2.4.1 Documentation must include all major maintenance activities such as contracted preventive maintenance and service and in-house activities such as the replacement of electrical components, lamps, tubing, valves, columns, detectors, cleaning and adjustments.

20.2.4.2 Each entry in the instrument log includes the Analyst's initials, date, a detailed description of the problem (or maintenance needed/scheduled), a detailed explanation of the solution or maintenance performed, and a verification that the equipment is functioning properly (state what was used to determine a return to control, e.g., CCV run on 'date' was acceptable, or instrument recalibrated on 'date' with acceptable verification, etc.) must also be documented in the instrument records.

20.2.4.3 When maintenance or repair is performed by an outside agency, service receipts detailing the service performed can be affixed into the logbooks adjacent to pages describing the maintenance performed. This stapled-in page must be signed across the page entered and the logbook, so it is clear that a page is missing if only half a signature is found in the logbook.

20.2.5 Instrument Repair

If an instrument requires repair (subjected to overloading or mishandling, gives suspect results, or otherwise has shown to be defective or outside of specified limits) it must be taken out of operation and tagged as out of service or otherwise isolated until such a time as the repairs have been made and the instrument can be demonstrated as operational by calibration and/or verification or other test to demonstrate acceptable performance. The laboratory must examine the effect of this defect on previous analyses.

20.2.6 Equipment Malfunction

In the event of equipment malfunction that cannot be resolved, service shall be obtained from the instrument vendor manufacturer, or qualified service technician, if such a service can be tendered. If on-site service is unavailable, arrangements shall be made to have the instrument shipped back to the manufacturer for repair. Backup instruments, which have been approved, for the analysis shall perform the analysis normally carried out by the malfunctioning instrument. If the backup is not available and the analysis cannot be carried out within the needed timeframe, the samples must be subcontracted.

20.2.7 Instrument Transfer or Send-Out

If an instrument is sent out for service or transferred to another facility, it must be recalibrated and verified (including new initial MDL study) prior to return to lab operations.

20.3 SUPPORT EQUIPMENT

This section applies to all devices that may not be the actual test instrument, but are necessary to support laboratory operations. These include but are not limited to balances, ovens, refrigerators, freezers, incubators, water baths, field sampling devices, temperature measuring devices, dispensing devices, if quantitative results are dependent on their accuracy, as in standard preparation and dispensing or dilution into a specified volume. All raw data records associated with the support equipment are retained to document instrument performance.

20.3.1 Weights and Balances

The accuracy of the balances used in the laboratory is checked every working day, before use. All balances are placed on stable counter tops.

Each balance is checked prior to initial serviceable use with at least two certified ASTM Type 1 weights spanning its range of use (weights that have been calibrated to ASTM Type 1 weights may also be used for daily verification). ASTM Type 1 weights used only for calibration of other weights (and no other purpose) are inspected for corrosion, damage or nicks at least annually and if no damage is observed. They are calibrated at least every five years by an outside calibration laboratory. Any weights (including ASTM Type 1) used for daily balance checks or other purposes are recalibrated/recertified annually to NIST standards (this may be done internally if laboratory maintains “calibration only” ASTM Type 1 weights).

All balances are serviced annually by a qualified service representative, who supplies the laboratory with a certificate that identifies traceability of the calibration to the NIST standards.

All of this information is recorded in logs, and the recalibration/recertification certificates are kept on file. Reference SOP NC-QA-015, Equipment Monitoring and Thermometer Calibration. A list of balances is in Table 21.2.

20.3.2 pH, Conductivity, and Turbidity Meters

The pH meters used in the laboratory are accurate to $\pm$ 0.1 pH units, and have a scale readability of at least 0.05 pH units. The meters automatically compensate for the temperature, and are calibrated with at least two working range buffer solutions before each use.

Conductivity meters are also calibrated before each use with a known standard to demonstrate the meters do not exceed an error of 1% or one umhos/cm.

Turbidity meters are also calibrated before each use. All of this information is documented in logs.

Consult pH and Conductivity, and Turbidity SOPs for further information.

20.3.3 Thermometers

All thermometers are calibrated on an annual basis with a NIST-traceable thermometer. IR thermometers, digital probes, and thermocouples are calibrated quarterly.

The NIST thermometer is recalibrated every three years (unless thermometer has been exposed to temperature extremes or apparent separation of internal liquid) by an approved outside service and the provided certificate of traceability is kept on file. The NIST thermometer(s)have increments of 1 degree (0.5 degree or less increments are required for drinking water microbiological laboratories), and have ranges applicable to method and certification requirements. The NIST traceable thermometer is used for no other purpose than to calibrate other thermometers.

All of this information is documented in logsheets. Monitoring method-specific temperatures, including incubators, heating blocks, water baths, and ovens, is documented in method-specific logsheets. More information on this subject can be found in SOP NC-QA-015, Equipment Monitoring and Thermometer Calibration.

20.3.4 Refrigerators/Freezer Units, Waterbaths, Ovens and Incubators

The temperatures of all refrigerator units and freezers used for sample and standard storage are monitored each working day (seven days a week for DOD labs).

Ovens, waterbaths and incubators are monitored on days of use.

All of this equipment has a unique identification number, and is assigned a unique thermometer for monitoring.

Sample storage refrigerator temperatures are kept between or 4 $\pm$ 2^{o}C.

Specific temperature settings/ranges for other refrigerators, ovens waterbaths, and incubators can be found in method specific SOPs.

All of this information is documented in Daily Temperature Logsheets posted on each unit and method-specific logbooks.

20.3.5 Autopipettors, Dilutors, and Syringes

Mechanical volumetric dispensing devices including burettes (except Class A Glassware) are given unique identification numbers and the deliveery volumes are verified gravimetrically, at a minimum, on a quarterly basis. Glass micro-syringes are considered the same as Class A glassware.

Micro-syringes are purchased from Hamilton Company. Each syringe is traceable to NIST. The laboratory keeps on file an “Accuracy and Precision Statement of Conformance” from Hamilton attesting established accuracy.

The laboratory maintains a sufficient inventory of autopipettors, and dilutors of differing capacities that fulfill all method requirements.

These devices are given unique identification numbers, and the delivery volumes are verified gravimetrically, at a minimum, on a quarterly basis.

Any device not regularly verified cannot be used for any quantitative measurements.

Micro-syringes are purchased from Hamilton Company. Each syringe is traceable to NIST. The laboratory keeps on file an “Accuracy and Precision Statement of Conformance” from Hamilton attesting established accuracy.

20.3.7 Field Sampling Devices (ISCO Autosamplers)

Each autosampler (ISCO) is assigned a unique identification number in order to keep track of the calibration. This number is recorded on the sampling documentation in a logbook.

The autosampler is calibrated semi-annually by setting the sample volume to 100ml and recording the volume received. The results are filed in a logbook/binder. The autosampler is programmed to run three cycles, and each of the three cycles is measured into a beaker to verify 100 ml are received.

If the RSD (Relative Standard Deviation) between the three cycles is greater than 20%, the procedure is repeated. If the result is still greater than 20%, the following options may be employed:

1) The unit is taken out of service.
2) The unit is used to pull composite samples only over a 24-hour period.

The results of this check are kept in a logbook/binder.

20.4 INSTRUMENT CALIBRATIONS

Calibration of analytical instrumentation is essential to the production of quality data. Strict calibration procedures are followed for each method. These procedures are designed to determine and document the method detection limits, the working range of the analytical instrumentation and any fluctuations that may occur from day to day.

Sufficient raw data records are retained to allow an outside party to reconstruct all facets of the initial calibration. Records contain, but are not limited to, the following: calibration date, method, instrument, analyst(s) initials or signatures, analysis date, analytes, concentration, response, type of calibration (Avg RF, curve, or other calculations that may be used to reduce instrument responses to concentration.)

Sample results must be quantitated from the initial calibration and may not be quantitated from any continuing instrument calibration verification unless otherwise required by regulation, method or program.

If the initial calibration results are outside of the acceptance criteria, action is performed and any affected samples are re-analyzed if possible. If the re-analysis is not possible, any data associated with an unacceptable initial calibration must be reported with appropriate data qualifiers (refer to Section 12). For the Ohio EPA Voluntary Action Program (VAP), please refer to the SOPs for the acceptable criteria, corrective actions, and exceptions.

Note: Instruments are calibrated initially and as needed after that and at least annually.

20.4.1 CALIBRATION STANDARDS

Calibration standards are prepared using the procedures indicated in the Reagents and Standards section of the determinative method SOP.

Standards for instrument calibration are obtained from a variety of sources. All standards are traceable to national or international standards of measurement, or to national or international standard reference materials.

The lowest concentration calibration standard that is analyzed during an initial calibration must be at or below the stated reporting limit for the method based on the final volume of extract (or sample).

The other concentrations define the working range of the instrument/method or correspond to the expected range of concentrations found in actual samples that are also within the working range of the instrument/method. Results of samples not bracketed by initial instrument calibration standards (within calibration range to 3 significant figures) must be reported as having less certainty, e.g., defined qualifiers or flags (additional information may be included in the case narrative). The exception to these rules is ICP methods or other methods where the referenced method does not specify two or more standards.

All initial calibrations are verified with a standard obtained from a second source and traceable to a national standard, when available (or vendor certified different lot if a second source is not available). For unique situations, such as air analysis where no other source or lot is available, a standard made by a different analyst would be considered a second source. This verification occurs immediately after the calibration curve has been analyzed, and before the analysis of any samples.

20.4.2 Calibration Verification

The calibration relationship established during the initial calibration must be verified at initially and at least daily as specified in the laboratory method SOPs in accordance with the referenced analytical methods and NELAC (2003) standard, Section 5.5.5.10. The process of calibration verification applies to both external standard and internal standard calibration techniques, as well as to linear and non-linear calibration models.

Note: The process of calibration verification referred to is fundamentally different from the approach called "calibration" in some methods. As described in those methods, the calibration factors or response factors calculated during calibration are used to update the calibration factors or response factors used for sample quantitation. This approach, while employed in other EPA programs, amounts to a daily single-point calibration.

All target analytes and surrogates, including those reported as non-detects, must be included in periodic calibration verifications for purposes of retention time confirmation and to demonstrate that calibration verification criteria are being met.

All samples must be bracketed by periodic analyses of standards that meet the QC acceptance criteria (e.g., calibration and retention time). The frequency is found in the determinataive methods or SOPs.

Note: If an internal standard calibration is being used (basically GCMS), then bracketing standards are not required. Only daily verifications are needed. The results from these verification standards must meet the calibration veirfication criteria and the retention time criteria (if applicable).

Generally, the initial calibrations must be verified at the beginning of each 12-hour analytical shift during which samples are analyzed. (Some methods may specify more or less frequent verifications). The 12-hour analytical shift begins with the injection of the calibration verification standard (or the MS tuning standard in MS methods). The shift ends after the completion of the analysis of the last sample or standard that can be injected within 12 hours of the beginning of the shift.

A continuing instrument calibration verification (CCV) must be repeated at the beginning and, for methods that have quantitation by external calibration models, at the end of each analytical batch. Some methods have more frequent CCV requirements see specific SOPs. Most Inorganic methods require the CCV to be analyzed after ever 10 samples or injections include matrix or batch QC samples.

Note: If an internal standard calibration is being used (basically GCMS) then bracketing standards are not required, only daily verifications are needed. The results from these verification standards must meet the calibration verification criteria and the retention time criteria (if applicable).

20.4.2.1 Verification of Linear Calibrations

Calibration verification for linear calibrations involves the calculation of the percent drift or the percent difference of the instrument response between the initial calibration and each subsequent analysis of the verification standard. (These calculations are available in the laboratory method SOPs.) Verification standards are evaluated based on the % Difference from the average CF or RF of the initial calibration or based on % Drift or % Recovery if a linear or quadratic curve is used.

Regardless of whether a linear or non-linear calibration model is used, if initial verification criterion is not met, then no sample analyses may take place until the calibration has been verified or a new initial calibration is performed that meets the specifications listed in the method SOPs. If the calibration cannot be verified after the analysis of a single verification standard, then adjust the instrument operating conditions and/or perform instrument maintenance, and analyze another aliquot of the verification standard. If the calibration cannot be verified with the second standard, then a new initial calibration is performed.

When the acceptance criteria for the calibration verification are exceeded high, i.e., high bias, and there are associated samples that are non-detects, then those non-detects may be reported. Otherwise, the samples affected by the unacceptable calibration verification shall be reanalyzed after a new calibration curve has been established, evaluated and accepted.

When the acceptance criteria for the calibration verification are exceeded low, i.e., low bias, those sample results may be reported if they exceed a maximum regulatory limit/decision level. Otherwise, the samples affected by the unacceptable verification shall be reanalyzed after a new calibration curve has been established, evaluated and accepted. For Ohio VAP samples, results may not be reported when calibration verifications are exceeded low.

20.5 TENTATIVELY IDENTIFIED COMPOUNDS (TICS) – GC/MS ANALYSIS

For samples containing components not associated with the calibration standards, a library search may be made for the purpose of tentative identification. The necessity to perform this

type of identification will be determined by the purpose of the analyses being conducted. Data system library search routines should not use normalization routines that would misrepresent the library or unknown spectra when compared to each other.

Note: If the TIC compound is not part of the client target analyte list but is calibrated by the laboratory and is both qualitatively and/or quantitatively identifiable, it should not be reported as a TIC. If the compound is reported on the same form as true TICs, it should be qualified and/or narrated that the reported compound is qualitatively and quantitatively (if verification in control) reported compared to a known standard that is in control (where applicable).

For example, the RCRA permit or waste delisting requirements may require the reporting of non-target analytes. Only after visual comparison of sample spectra with the nearest library searches may the analyst assign a tentative identification.

20.6 GC/MS TUNING

Prior to any GCMS analytical sequence, including calibration, the instrument parameters for the tune and subsequent sample analyses within that sequence must be set.

Prior to tuning/auto-tuning the mass spec, the parameters may be adjusted within the specifications set by the manufacturer or the analytical method. These generally don't need any adjustment but it may be required based on the current instrument performance. If the tune verification does not pass it may be necessary to clean the source or perform additional maintenance. Any maintenance is documented in the maintenance log.

Table 20-1. Laboratory Equipment and Instrumentation

Instrument Type	Manufacturer	Model/Serial No.	Year into Service
GC/MS Volatiles	Hewlett-Packard (UX2)	5971A-5890, S/N 3140A38490	1992
	Hewlett-Packard (HP6)	5973-6890, S/N US00005571	2006
	Hewlett-Packard (UX7)	5973-6890, S/N US00010937	1998
	Hewlett-Packard (UX8)	5973-6890, S/N US00027773	1999
	Hewlett-Packard (UX9)	5973-6890, S/N US00028329	2000
	Hewlett-Packard (UX10)	5973-6890, S/N US00032072	2000
	Agilent (UX11)	5973-6890, S/N US00038093	2000
	Agilent (UX12)	5973-6890, S/N US10202133	2002
	Agilent (UX14)	5973-6890, S/N CN10340027	2003
	Agilent (UX15)	5973-6890, S/N CN10515062	2005
	Agilent (UX16)	5975-6890, S/N CN10539065	2005
GC/MS Volatiles Autosampler	OI Analytical (UX2)	4552	1999
	OI Analytical (HP6)	4552	1998
	OI Analytical (UX7)	4552	1998
	OI Analytical (UX8)	4552	1999
	OI Analytical (UX9)	4552	2000
	OI Analytical (UX10)	4552	2000
	OI Analytical (UX11)	4552	2000
	OI Analytical (UX12)	4552	2002
	OI Analytical (UX14)	4552	2003
	OI Analytical (UX15)	4552	2005
	OI Analytical (UX16)	4552	2005
GC/MS Volatiles Purge and Trap	OI Analytical (UX2)	4560	1999
	OI Analytical (HP6)	Encon	1998
	OI Analytical (UX7)	4660	2004
	OI Analytical (UX8)	4560	2004
	OI Analytical (UX9)	4560	2000
	OI Analytical (UX10)	4660	2003
	OI Analytical (UX11)	4560	2000
	OI Analytical (UX12)	4560	2002
	OI Analytical (UX14)	4560	2003
	OI Analytical (UX15)	4660	2005

Instrument Type	Manufacturer	Model/Serial No.	Year into Service
	OI Analytical (UX16)	4660	2005
	EST (spare)	4560	2002
GC/MS Semivolatiles	Hewlett-Packard HP7	5973-6890, S/N US00009247	1998
	Hewlett-Packard HP8	5973-6890, S/N US00022627	1999
	Hewlett-Packard HP9	5973-6890, S/N US00027943	2000
	Agilent HP10	5973-6890, S/N CN10340002	2003
	Agilent A4AG2	5975-C, S/N CN10721110	2007
GC Volatiles	Agilent (A)	6890 PID/FID, S/N US10402056	2004
	Hewlett-Packard (O)	6890 Dual PID/Hall, S/N US00007206	1997
	Hewlett-Packard (P)	6890 PID/HALL, S/N US00030616	1997
	Hewlett-Packard (Y)	6890N PID/FID, S/N US10337062	2003
	Agilent (Z)	6890 EPC & PDD/FID, S/N 10205072	2000
	Agilent (N)	7890 Atomic Fluorescence, S/N CN10820009	2008
GC Volatiles Auto Sampler	OI Analytical (O)	Archon	2000
	OI Analytical (Y)	4552	1998
	Varian (A)	Archon	1998
	Varian (P)	4552	2000
	Agilent (Z)	7694	2000
	EST (N)	Centurion	2008
GC Volatiles Purge & Trap	Tekmar (O)	4560	2000
	Tekmar (P)	3000	1993
	OI Analytical (A)	4560	1998
	OI Analytical (Y)	3000	1993
	Tekmar (N)	Stratum	2008
GC Semivolatiles	Hewlett-Packard (P1)	6890 EPC & Dual ECD Y-Splitter S/N US00023208	1998
	Hewlett-Packard (P2)	6890 EPC & Dual ECD Y-Splitter S/N US00023512	1998
	Hewlett-Packard (P3)	6890 EPC & Dual ECD Y-Splitter S/N US00023674	1998
	Hewlett-Packard (P4)	6890 EPC & Dual ECD Y-Splitter S/N US00029531	1999
	Hewlett-Packard (P6)	6890 EPC & Dual FID S/N US00032848	2000

Instrument Type	Manufacturer	Model/Serial No.	Year into Service
	Agilent (P7)	6890N EPC & Dual FID S/N US10202132	2000
	Agilent (P9)	6890N EPC & Dual ECD Y-Splitter S/N US10205045	2005
	Agilent (P10)	6890 EPC & Dual ECD Y-Splitter S/N US10151110	1999
	Agilent (P11)	6890N EPC & Dual ECD Y-Splitter S/N CN10517088	2004
	Agilent (P12)	6890N EPC & Dual ECD Y-Splitter S/N CN10512025	2005
	Agilent (P13)	6890N EPC & Dual ECD Y-Splitter S/N CN10435032	2004
GC Semivolatiles HPLC	Hewlett-Packard (L2)	HPLC 1100, S/N US82404153	1998
Extractions Sonicators	Misonix	3000 (self-tuning), S/N R1044	2005
	Misonex	Ultrasonic Processor XL, S/N G4221	
	Heat Systems	XL2020, S/N G1026	2001
Extractions 6-Position Accelerated Soxhlet Extractor	Gerhardt Soxtherm	8 units	2003
Extractions pH Meter	Thermo Orion	420, S/N 074028	1998
	Mettler Toledo	SevenEasy pH (self-calibrating) S/N 1228295055	2008
	Denver Instrument (spare)	UB-5 S/N UB-5093011	2004
Metals ICP	Thermo Jarrell Ash (I-5)	Trace Analyzer 61E, S/N 273490	1994
	Thermo Jarrell Ash (I-6)	Trace Analyzer 61E, S/N 269490	1994
Metals ICP/MS	Thermo (I-8)	Series 2, S/N 01137C	2007
Metals Mercury Analyzer	Leeman (CVAA) (H1)	PS200 II, S/N HG9031	1999
	Leeman (CVAF) Low Level (H3)	Hydra AF Gold+, Model #112-00067-1 S/N AFG1006	2001
	Leeman (CVAA) (H4)	Hydra AA , S/N 6011	2006
WC Arsenic Speciation	Trace Detect	Nano-Band Explorer, S/N NBE00017	2003
WC Autotitrator	Man-Tech	PC – Titrate, S/N MS-9K8-217	2001

Instrument Type	Manufacturer	Model/Serial No.	Year into Service
WC Block Digester	Andrews	2210 Phenol	1999
	Andrews	2205 Ammonia	1999
	Lachat	BD46 TKN	1992
WC BOD	Mantech	BOD, S/N N817947	1999
WC Conductivity	Man-Tech	4310, S/N 1613	1989
WC Cyanide	LabCrest MidiDist	PRG-2520-BL, S/N 1000-99-01	1999
WC Discrete Analyzer	Kone	Konelab 200, Z1718383	2001
	Kone	Konelab 250, A2120021	2005
WC Dissolved Oxygen Meter	YSI	52C E, S/N 99C1094	1993
WC Flashpoint	Herzog	HFP 339, S/N 073390084	2007
	Petrolab (199443)	Petrotest, S/N 0741990603	1999
WC Ion Chromatograph	Dionex	DX-320, S/N 00060187	2001
	Dionex	DX-120, S/N 98110093	1999
WC pH Meter	Orion pH Meter	320, S/N 020032	2007
	Orion (Ammonia ISE)	520A, S/N 48029	1996
WC Residual Chlorine Meter	Hanna	HI 93701	2000
	Hanna	HI 93701	2006
WC TOC	OI Analytical	1010 TOC Analyzer, S/N K503710931	2005
WC TOX	Thermo Electron	1200, S/N 973515	1998
	Thermo Electron	1200, S/N 2001.0174	2005
	Thermo Electron	1200, S/N 2005.0234	2005
WC TRAACS	Bran & Luebbe	800, S/N 890140	1993
WC Turbidimeter	HF Scientific	Micro 100, S/N 200705143	2001
WC UV / VIS	Milton Roy	Spectronic 401, S/N 381A170002	1993
	Genesys	Spectronic 20, S/N 3SGL078016	1998
	Genesys	Spectronic 20, S/N 3SCA174040	2008
	Genesys	Spectronic 20, S/N 3SGL226006 (Model 4001/4)	2008
WC Sulfide	Westco EasyDist		2008

Table 20-2. Schedule of Routine Maintenance
(Refer to manufacturer's instructions for each instrument to identify and perform maintenance operations)

INSTRUMENT MAINTENANCE SCHEDULE
ION CHROMATOGRAPH

As Needed	Daily	Weekly	Monthly
Clean micro-membrane suppressor when decreases in sensitivity are observed.	Check plumbing/leaks.	Check pump heads for leaks.	Check all air and liquid lines for discoloration and crimping, if indicated.
Check fuses when power problems occur.	Check gases.	Check filter (inlet)	Check/change bed supports guard and analytical columns, if indicated.
Reactivate or change column when peak shape and resolution deteriorate or when retention time shortening indicates that exchange sites have become deactivated.	Check pump pressure.		
De-gas pump head when flow is erratic.	Check conductivity meter.		

INSTRUMENT MAINTENANCE SCHEDULE
TOTAL ORGANIC HALIDE ANALYZER

Daily	**As Needed**
Check electrodes for damage; polish the electrodes.	Examine and clean or replace pyrolysis tube.
Replace dehydrating fluid and electrolyte fluid.	Clean titration cell.
Clean quartz boat.	Observe gas flow.
Observe check valves during use for backfeed.	Replace reference electrode fluid.
At end of each day of use, wash out absorption module, empty electrolyte and fill cell with DI water. Empty dehydrator tube	Change quartz wool.
Perform cell performance check.	Replace O-rings and seals.

INSTRUMENT MAINTENANCE SCHEDULE
HIGH PRESSURE LIQUID CHROMATOGRAPH

Daily	**As Needed**
Check level of solution in reservoirs. If adding, verify that solvent is from the same source. If changing, rinse gas and delivery lines to prevent contamination of the new solvent.	Replace columns when peak shape and resolution indicate that chromatographic performance of column is below method requirements.
Check gas supply.	Oil autosampler slides when sample does not advance.
Flush with an appropriate solvent to remove all bubbles.	Rinse flow cell with 1N nitric acid if sensitivity low.
Pre-filter all samples.	Change pump seals when flow becomes inconsistent.
	Repack front end of column Backflush column.

INSTRUMENT MAINTENANCE SCHEDULE
ICP AND ICP/MS

Daily	Monthly or As Needed	Semi-Annually	Annually
Check vacuum pump gage. (<10 millitorr)	Clean plasma torch assembly to remove accumulated deposits	Change vacuum pump oil	Notify manufacturer service engineer for scheduled preventive maintenance service
Check cooling water supply system is full and drain bottle is not full. Also drain tubing is clear, tight fitting, and has few bends.	Clean nebulizer and drain chamber; keep free flowing to maintain optimum performance	Replace coolant water filter (may require more or less frequently depending on quality of water)	
Check nebulizer is not clogged	Clean filters on back of power unit to remove dust		
Check capillary tubing is clean and in good condition	Replace when needed: - peristaltic pump tubing - sample capillary tubing - autosampler sipper probe		
Check peristaltic pump windings are secure	- Check yttrium position - Check O-rings - Clean/lubricate pump rollers		
Check high voltage switch is on			
Check torch, glassware, aerosol injector tube, and bonnet are clean			

INSTRUMENT MAINTENANCE SCHEDULE
CVAS AND CVAFS

Daily	As Needed	Annually
Change drying tube	Change pump tubing	Change Hg lamp
Check pump tubing/drain tubing	Check/change Hg lamp	
Check gas pressure	Clean optical cell	
Check aperture reading Check tubing	Lubricate pump	

INSTRUMENT MAINTENANCE SCHEDULE
GAS CHROMATOGRAPH

Daily *	**As Needed**
Check for sufficient supply of carrier and detector gases. Check for correct column flow and/or inlet pressures.	Replace front portion of column packing or break off front portion of capillary columns. Replace column if this fails to restore column performance, or when column performance (e.g., peak tailing, poor resolution, high backgrounds, etc.) indicates it is required. Quarterly FID: clean detector, only as needed—not quarterly/or semi-annually.
Check temperatures of injectors and detectors. Verify temperature programs by RT shift.	Change glass wool plug in injection port and/or replace injection port liner when front portion of column packing is changed or front portion of capillary column is removed.
Clean injector port weekly for TPH for 8015B, when breakdown fails; otherwise, when RT shift or bad samples run.	Annually FID: replace flame tip, only as needed. Only as needed: ECD--detector cleaning and re-foiling, whenever loss of sensitivity, erratic response, or failing resolution is observed
Check baseline level during analysis of run—not maintenance.	Perform gas purity check (if high baseline indicates that impure carrier gas may be in use).
Watched weekly: check reactor temperature of electrolytic conductivity detector. Inspect chromatogram to verify symmetrical peak shape and adequate resolution between closely eluting peaks, when analyzing pesticides; part of analysis—not maintenance. Clip column leader when chromatography looks bad—not daily.	Replace or repair flow controller if constant gas flow cannot be maintained. Replace fuse. Reactivate external carrier gas dryers. Detectors: clean when baseline indicates contamination or when response is low. FID: clean/replace jet, replace ignitor. ECD: follow manufacturer's suggested maintenance schedule. Reactivate flow controller filter dryers when presence of moisture is suspected. HP 7673 Autosampler: replace syringe, fill wash bottle, dispose of waste bottle contents.

*No daily maintenance done on any instrument/method. Weekly change IPL on TPH instrument. Everything else is on an "as needed" basis.

INSTRUMENT MAINTENANCE SCHEDULE
MASS SPECTROMETER

Daily	Weekly	As Needed	Quarterly	Annually
Check for sufficient gas supply. Check for correct column flow and/or inlet pressure.	Check mass calibration (PFTBA or FC-43)	Check level of oil in mechanical pumps and diffusion pump if vacuum is insufficient. Add oil if needed between maintenance.	Check ion source and analyzer (clean, replace parts as needed)	Replace the exhaust filters on the mechanical rough pump every 1-2 years.
Check temperatures of injector, detector. Verify temperature programs.		Replace electron multiplier when the tuning voltage approaches the maximum and/or when sensitivity falls below required levels.	Check vacuum, relays, gas pressures and flows	
Check inlets, septa		Clean Source, including all ceramics and lenses - the source cleaning is indicated by a variety of symptoms including inability of the analyst to tune the instrument to specifications, poor response, and high background contamination.	Change oil in the mechanical rough pump.	
Check baseline level		Repair/replace jet separator.		
Check values of lens voltages, electron multiplier, and relative abundance and mass assignments of the calibration compounds.		Replace filaments when both filaments burn out or performance indicates need for replacement.		

INSTRUMENT MAINTENANCE SCHEDULE
TRAACS AUTO ANALYZER

As Needed	Daily
Replaces air filter when progressive loss of air pressure is observed.	Check air pressure gauge (22 ± 2 psi)
Replace air valve tubing when occlusion in tubing is observed	Use recommended washout procedure (at end of analysis operations)
Change all pump tubes (or after 200 hours of pumping time, or after 1000 hours of pumping time)	
Clean sample probe shaft	
Replace pump platens	
Lightly lubricate the linear sample rails (use semi-fluid lubricant)	
Replace colorimeter lamp (or after 2500 hours of use)	

INSTRUMENT MAINTENANCE SCHEDULE
ANALYTICAL/TOP LOADING BALANCES

Daily	Annually
Check using Class 1-verified weights once daily or before use Clean pan and weighing compartment	Manufacturer cleaning and calibration

INSTRUMENT MAINTENANCE SCHEDULE
REFRIGERATORS/WALK-IN COOLERS

Daily	As Needed
Temperatures checked and logged	Refrigerant system and electronics serviced

INSTRUMENT MAINTENANCE SCHEDULE
OVENS

Daily	As Needed
Temperatures checked and logged	Electronics serviced

INSTRUMENT MAINTENANCE SCHEDULE
SPECIFIC DIGITAL ION ANALYZER

Daily	As Needed
Daily when used: • Calibrate with check standards • Inspect electrode daily, clean as needed • Inspect electrode proper levels of filling solutions daily; fill as needed • Clean probe after each use	Electronics serviced

INSTRUMENT MAINTENANCE SCHEDULE
TURBIDIMETER

Daily	Monthly	As Needed
Daily when used: • Adjust linearity on varying levels of NTU standards. Standardize with NTU standards • Inspect cells	Clean instrument housing	Electronics serviced

INSTRUMENT MAINTENANCE SCHEDULE
DISSOLVED OXYGEN METER

Daily	As Needed
Daily when used: • Calibrate with saturated air • Check probe membrane for deterioration • Clean and replace membrane with HCl solution	• Electronics serviced • Clean and replace membrane with HCl solution

INSTRUMENT MAINTENANCE SCHEDULE
CONDUCTANCE METER

Daily	As Needed
Daily when used: • Check probe and cables • Inspect conductivity cell	Electronics serviced

INSTRUMENT MAINTENANCE SCHEDULE
CHEMICAL OXYGEN DEMAND (COD) REACTOR [1]

Daily	As Needed
Daily when used: • Calibrate with check standards	Electronics serviced

INSTRUMENT MAINTENANCE SCHEDULE
SPECTROPHOTOMETER

As Needed	Daily	Monthly	Annually
Dust the lamp and front of the front lens	Check the zero % adjustment	Clean windows	Check instrument manual
	Clean sample compartment		Perform wavelength calibration
	Clean cuvettes		Replace lamp annually or when erratic response is observed
			Clean and align optics

INSTRUMENT MAINTENANCE SCHEDULE
pH METER

As Needed	Daily
Clean electrode	Inspect electrode. Verify electrodes are properly connected and filled
Refill reference electrode	Inspect electrode proper levels of filling solutions. Make sure electrode is stored in buffer

INSTRUMENT MAINTENANCE SCHEDULE
TOTAL ORGANIC CARBON ANALYZER

Daily	As Needed	Weekly	Monthly
Check: • Oxygen supply • Persulfate supply • Acid supply • Carrier gas flow rate (~ 150 cc/min) • IR millivolts for stability (after 30 min. warm-up) • Reagent reservoirs	Check injection port septum after 50-200 runs Tube end-fitting connections after 100 hours or use Indicating drying tube NDIR zero, after 100 hours of use Sample pump, after 2000 hours for use Digestion vessel/condensation chamber, after 2000 hours of use Permeation tube, after 2000 hours of use NDIR cell, after 2000 hours of use Change pump tubing	Check liquid-flow-rate-pump-tubing conditions on autosampler Check injection port septum	Clean digestion vessel Clean condenser column Do the leak test

Instrument Maintenance Schedule
Digestion Block

Annually
Check temperature with NIST thermometer

Instrument Maintenance Schedule
Flash Point Tester

Daily
Check tubing Clean sample cup each use
Check gas
Clean flash assembly
Check stirrer

Table 20-3. Preventive Maintenance Procedures

SUMMARY OF INORGANIC METHOD CALIBRATIONS

Analysis	Calibration	NPDES [1]		RCRA (SW846) [2]	
		Method	Requirement	Method	Requirement
Acidity	Initial	350.1	Two-level calibration that bracket the expected pH of the sample ± 0.05 pH units of true value	9040B 9045C	2 point calibration ± 0.05 pH units of true value
	Continuing	350.1	One buffer check every 10 samples ± 5% of true value	9040B 9045C	N/A
	Other	350.1	Third point check	9040B 9045C	Third point check
	Ending	350.1	One buffer check ± 5% of true value	9040B 9045C	N/A
Alkalinity, Bicarbonate, Carbonate	Initial	310.1 2320B	2 point calibration of pH meter ± 0.05 pH units of true value	--	N/A
	Continuing	310.1 2320B	N/A	--	N/A
	Ending	310.1 2320B	N/A	--	N/A
Ammonia	Initial	350.1	6 levels including blank, "r" [3] ≥ 0.995	--	N/A
	Continuing	350.1	One level or LCS every 10 samples ± 10% of true value	--	N/A
	Ending	350.1	One level or LCS every 10 samples ± 10% of true value	--	N/A
Arsenic Speciation		N/A	N/A	7063	* Refer to Section 10 of SOP NC-WC-0090

Analysis	Calibration	NPDES [1]		RCRA (SW846) [2]	
		Method	Requirement	Method	Requirement
Biochemical Oxygen Demand (BOD)	Initial	405.1 SM5210B	a. Winkler titration: Iodometric with standard thiosulfate b. Membrane electrode: Read in air and in water with zero dissolved oxygen	--	N/A
	Continuing	405.1 SM5210B	N/A	--	N/A
	Ending	405.1 SM5210B	N/A	--	N/A
Bromide	Initial	300.0A	5 levels plus a blank, "r"[3] ≥ 0.995	9056A	5 levels plus a blank, "r" [3] ≥ 0.995
	Continuing	300.0A	Level every 10 samples ± 10% of true value	9056A	N/A
	Ending	300.0A	N/A	9056A	N/A
Cation Exchange	Initial	N/A	N/A	9081	N/A
	Continuing	N/A	N/A	9081	N/A
	Ending	N/A	N/A	9081	N/A
Chemical Oxygen Demand (COD)	Initial	410.4 SM5220D	5 levels plus a blank"r" [3] ≥ 0.995	--	N/A
	Continuing	410.4 SM5220D	One level every 10 samples ± 10% of true value	--	N/A
	Ending	410.4 SM5220D	One level ± 10% of true value	--	N/A

Analysis	Calibration	NPDES [1]		RCRA (SW846) [2]	
		Method	Requirement	Method	Requirement
Chloride	Initial	300.0A 325.2	5 levels plus blank "r" [3] ≥ 0.995	9056A 9251	Method 9056: 3 levels plus a blank Method 9252: 5 levels plus blank "r" [3] ≥ 0.995
	Continuing	300.0A 325.2	One level every 10 samples ± 10% of true value	9056A 9251	Method 9056: One per batch of 20 samples, ± 10% of true value Method 9252: One level every 10 samples, ± 10% of true value
	Ending	300.0A 325.2	One level every 10 samples ± 10% of true value	9056A 9251	Method 9056 : N/A Method 9252: One level ± 10% of true value
Chromium Cr^{+6}	Initial	3500 Cr-D	3 levels plus blank	7196A	5 levels plus blank "r" [3] ≥ 0.995
	Continuing	3500 Cr-D	One level every 10 samples ± 10% of true value	7196A	One level every 10 samples ± 15%
	Ending	3500 Cr-D	One level ± 10% of true value	7196A	One level ± 15%
Chlorine, Residual	Initial	330.5 SM3500CL-G	N/A	--	N/A
	Continuing	330.5 SM3500CL-G	N/A	--	N/A
	Ending	330.5 SM3500CL-G	N/A	--	N/A

Analysis	Calibration	NPDES [1]		RCRA (SW846) [2]	
		Method	Requirement	Method	Requirement
Conductivity	Initial	120.1 SM2510B	Standard KCl solution	9050A	One level to determine cell constant
	Continuing	120.1 SM2510B	N/A	9050A	N/A
	Ending	120.1 SM2510B	N/A	9050A	N/A
Cyanide (Amenable)	Initial	335.1 SM4500CN-G	6 levels plus blank "r" [3] ≥ 0.995	9012A	6 levels plus blank "r" [3] ≥ 0.995
	Continuing	335.1 SM4500CN-G	One level every 10 samples ± 10% of true	9012A	One mid-level every 10 samples ± 15% of true value
	Ending	335.1 SM4500CN-G	One level ± 10 % of true value	9012A	± 15% of true value
Cyanide (Total)	Initial	335.1/335.2 335.3/335.4 SM4500CU-E/ 335.2-CLP-M	6 levels plus blank "r" [3] ≥ 0.995	9012A	6 levels plus blank "r" [3] ≥ 0.995
	Continuing	335.1/335.2 335.3/335.4 SM4500CU-E/ 335.2-CLP-M	One mid-level every 10 samples ± 10 % of true value	9012A	One mid-level every 10 samples ± 15% of true value
	Ending	335.1/335.2 335.3/335.4 SM4500CU-E/ 335.2-CLP-M	One mid-level ± 10 % of true value	9012A	± 15% of true value
Flashpoint	Initial	--	N/A	1010 ASTM D93-9	p-Xylene reference standard must have flashpoint of 81°F ±2°F
	Continuing	--	N/A	1010 ASTM D93-9	N/A
	Ending	--	N/A	1010 ASTM D93-9	N/A

Analysis	Calibration	NPDES [1]		RCRA (SW846) [2]	
		Method	Requirement	Method	Requirement
Fluoride	Initial	300.0A 340.2	Method 300.0A: 5 levels plus a blank, "r" [3] ≥ 0.995 Method 340.2: 5 levels "r" [3] ≥ 0.995	9056A	3 levels plus a blank
	Continuing	300.0A 340.2	One mid-level every 10 samples ± 10% of true value	9056A	One per batch of 20 samples ± 10% of true value
	Ending	300.0A 340.2	One mid-level ± 10% of true value	9056A	N/A
Hardness	Initial	130.2 2340B	Method 130.2: Standardize titrant Method 2340B: See ICP Metals 200.7	--	N/A
	Continuing	130.2 2340B	Method 130.2: N/A Method 2340B: See ICP Metals 200.7	--	N/A
	Ending	130.2 2340B	Method 130.2: N/A Method 2340B: See ICP Metals 200.7	--	N/A
Iron (Ferrous)	Initial	3500-Fe D	3 levels plus a blank, "r" [3] ≥ 0.995	-	N/A
	Continuing	3500-Fe D	One mid-level every 10 samples ± 10% of true value	-	N/A
	Ending	3500-Fe D	One mid-level ± 10% of true value	-	N/A

Analysis	Calibration	NPDES [1]		RCRA (SW846) [2]	
		Method	Requirement	Method	Requirement
Nitrate	Initial	300.0A 353.2 SM4500NO_3-E	5 levels plus a blank "r" [3] ≥ 0.995	9056A	3 levels plus a blank
	Continuing	300.0A 353.2 SM4500NO_3-E	One mid-level every 10 samples ± 10% of true value	9056A	One per batch of 20 samples ± 10% of true value
	Ending	300.0A 353.2 SM4500NO_3-E	One mid-level ± 10% of true value	9056A	N/A
Nitrite	Initial	300.0A 354.1	5 levels plus a blank "r" [3] ≥ 0.995	9056A	3 levels plus a blank
	Continuing	300.0A 354.1	One mid-level every 10 samples ± 10% of true value	9056A	One per batch of 20 samples ± 10% of true value
	Ending	300.0A 354.1	One mid-level ± 10% of true value	9056A	N/A
Nitrate-Nitrite	Initial	300.0A 353.2	5 levels plus blank "r" [3] ≥ 0.995	--	N/A
	Continuing	300.0A 353.2	One level every 10 samples ± 10% of true value	--	N/A
	Ending	300.0A 353.2	One mid-level ± 10% of true value	--	N/A

Analysis	Calibration	NPDES [1]		RCRA (SW846) [2]	
		Method	Requirement	Method	Requirement
Phosphorus (Total and Ortho-phosphate)	Initial	300.0A 365.1 SM4500P-E	Method 300.0A/365.3: 3 levels plus a blank Method 365.2: 5 levels plus a blank	--	N/A
	Continuing	300.0A 365.1 SM4500P-E	Method 300.0A/365.3: One level every 10 samples ± 10% of true value Method 365.2: Blank and 2 standards with each series of samples, ± 2% of true value or recalibrate	--	N/A
	Ending	300.0A 365.1 SM4500P-E	Method 300.0A/365.3: ± 10% of true value Method 365.2: N/A	--	N/A
pH	Initial	150.1 SM4500H-B	2 level calibration that bracket the expected pH of the sample ± 0.05 pH units of true value	9040B 9045C	2 point calibration ± 0.05 pH units of true value
	Continuing	150.1 SM4500H-B	One buffer check every 10 samples ± 5% of true value	9040B 9045C	N/A
	Other	150.1 SM4500H-B	Third point check	9040B 9045C	Third point check
	Ending	150.1 SM4500H-B	One buffer check ± 5% of true value	9040B 9045C	N/A

Analysis	Calibration	NPDES [1]		RCRA (SW846) [2]	
		Method	Requirement	Method	Requirement
Phenolics	Initial	420.1	5 levels plus a blank "r" [3] ≥ 0.995	9065 9066	5 levels plus a blank "r" [3] 0.995
	Continuing	420.1	One mid-level every 10 samples ± 10% true value	9065 9066	One mid-level ± 15% true value
	Ending	420.1	One mid-level ± 10% true value	9065 9066	One mid-level ± 15% true value
Phosphate	Initial	SM4500P-E	N/A	9056	3 levels plus a blank
	Continuing	SM4500P-E	N/A	9056	One per batch of 20 samples ± 15% of true value
	Ending	SM4500P-E	N/A	9056	N/A
Settleable Solids	Initial	160.5	N/A	--	N/A
	Continuing	160.5	N/A	--	N/A
	Ending	160.5	N/A	--	N/A
Specific Conductance	Initial	120.1 SM2510B	Standardize meter with 0.01 M KCl	9050A	N/A
	Continuing	120.1 SM2510B	One level every 10 samples ± 10% of true value	9050A	N/A
	Ending	120.1 SM2510B	One level ± 10% of true value	9050A	N/A
Sulfate	Initial	300.0A 375.4	Method 300.0A: 5 levels plus blank "r" [3] ≥ 0.995 Method 375.4: 3 levels plus blank "r" [3] ≥ 0.995	9038 9056A	Method 9038: 3 levels plus a blank for every hour of continuous sample analysis. Method 9056: 3 levels plus a blank

Analysis	Calibration	NPDES [1]		RCRA (SW846) [2]	
		Method	Requirement	Method	Requirement
Sulfate (Cont'd)	Continuing	300.0A 375.4	Method 300.0A: One mid-level after every 10 samples ± 10% of true value Method 375.4: One level every 3 or 4 samples ± 10% of true value	9038 9056A	Method 9038: Independent-prepared check standard every 15 samples Method 9056: 1 per batch of 20 samples ± 10% of true value
	Ending	300.0A 375.4	± 10% of true value	9038 9056A	N/A
Sulfide	Initial	376.1	Method 376.1: This is a titration method. Therefore, calibrations are not applicable.	9030B/ 9034 9030A	This is a colorimetric titration. Therefore, calibration is not applicable.
	Continuing	376.1	Method 376.1: N/A	9030B/ 9034 9030A	This is a colorimetric titration. Therefore, calibration is not applicable.
	Ending	376.1	Method 376.1: N/A	9030B/ 9034 9030A	This is a colorimetric titration. Therefore, calibration is not applicable.
Total Dissolved Solids	Initial	160.1 SM2540E	This is a gravimetric determination. Calibrate balance prior to analysis	--	N/A
	Continuing	160.1 SM2540E		--	N/A
	Ending	160.1 SM2540E		--	N/A

Analysis	Calibration	NPDES [1]		RCRA (SW846) [2]	
		Method	Requirement	Method	Requirement
Total Kjeldahl Nitrogen (TKN)	Initial	351.3 $SM4500NO_3$	Method 351.3: Titrimetric: Standardize titrant Colorimetric: 7 levels plus blank	--	N/A
	Continuing	351.3 $SM4500NO_3$	Method 351.3: N/A	--	N/A
	Ending	351.3 $SM4500NO_3$	Method 351.3: N/A	--	N/A
Total Organic Carbon (TOC)	Initial	415.1 SM5310D	3 levels plus blank	9060 Walkley Black	3 levels plus blank "r" [3] ≥ 0.995
	Continuing	415.1 SM5310D	1 mid-level every 10 samples ± 15% of true value	9060 Walkley Black	1 mid-level every 10 samples ± 15% of true value
	Ending	415.1 SM5310D	± 15% of true value	9060 Walkley Black	± 15% of true value
Total Organic Halides (TOX)	Initial	450.1	Method 450.1: Daily instrument calibration standard and blank in duplicate ± 10% of true value (calibration standard) Verify with independently-prepared check standard	9020B 9023 (EOX)	Daily instrument calibration standard and blank in duplicate ± 10% of true value (calibration standard) Verify with independently-prepared check standard –ICV ± 10% SOP CORP-WC-0001

Analysis	Calibration	NPDES [1]		RCRA (SW846) [2]	
		Method	Requirement	Method	Requirement
Total Organic Halides (TOX) (cont'd)	Continuing	450.1	± 10% of true value	9020B 9023 (EOX)	CCV ± 10% of true value SOP CORP-WC-0001
	Ending	450.1	± 10% of true value	9020B 9023 (EOX)	CCV ± 10% of true value SOP CORP-WC-0001
Total Solids	Initial	160.3	This is a gravimetric determination. Calibrate balance before use.	--	N/A
	Continuing	160.3		--	N/A
	Ending	160.3		--	N/A
Total Suspended Solids (Nonfilterable)	Initial	160.2 SM2540E	This is a gravimetric determination. Calibrate balance before use.	--	N/A
	Continuing	160.2 SM2540E		--	N/A
	Ending	160.2 SM2540E		--	N/A
Turbidity	Initial	180.1	Minimum of 1 level in each instrument range. Follow manufacturer's instructions	--	N/A
	Continuing	180.1	N/A	--	N/A
	Ending	180.1	N/A	--	N/A
Volatile Solids	Initial	160.4	This is a gravimetric determination. Calibrate balance before use.	--	N/A
	Continuing	160.4		--	N/A
	Ending	160.4		--	N/A

Analysis	Calibration	NPDES [1]		RCRA (SW846) [2]	
		Method	Requirement	Method	Requirement
ICP & ICP/MS Metals (excludes Hg)	Initial	200.7	One level and blank. ICV RSD <3% from replicate - daily	6010B	One level and blank. ICV RSD <5% from replicate - daily
	Initial	200.8	One level and blank	6020	One level and blank
	Continuing	200.7	Every 10 samples ±10% of true value CCV RSD < 5% from replicate	6010B	Mid-level calibration standard Every 10 samples ± 10% of true value CCV RSD < 5% from replicate
	Continuing	200.8	N/A	6020	N/A
	Ending	200.7	±10% of true value CCV RSD < 5% from replicate	6010B	Mid-level calibration standard ± 10% of true value CCV RSD < 5% from replicate
	Ending	200.8	N/A	6020	N/A
	Other	200.7	ICSA, ICSAB: Analyze at beginning of run. For ICSA, AB criteria see SOP Semi-Annually: ICP interelement correction factors Instrument detection limits	6010B	ICSA, ICSAB: Analyze at beginning of run. For ICSA, AB criteria see SOP Semi-Annually: ICP interelement correction factors Instrument detection limits
	Other	200.8	N/A	6020	N/A

Analysis	Calibration	NPDES [1]		RCRA (SW846) [2]	
		Method	Requirement	Method	Requirement
Mercury by CVAA & CVAFS	Initial	245.1 1631E	5 levels plus blank ICV ±10% of true value "r" [3] ≥ 0.995	7470A 7471A	5 levels plus blank ICV ± 10% of true value "r" [3] ≥ 0.995
	Continuing	245.1* 1631E	Daily or every 10 samples, whichever is more frequent ±20% of true value	7470A 7471A	Every 10 samples ±20% of true value
	Ending	245.1 1631E	±20% of true value	7470A 7471A	±20% of original prepared standard
	Other	245.1 1631E	Annually: MDL	7470A 7471A	Annually: MDL

* 245.1 continuing – Initial CCV ±5% of true value

Footnotes

[1] National Pollutant Discharge Elimination System.

[2] Resource Conservation and Recovery Act, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, (SW-846), Third Edition, September 1986. Contains Final Update I (July 1992), Final Update IIA (August 1993), Final Update II (September 1994), Final Update IIB (January 1995), and Final Update III (December, 1996).

[3] "r" = correlation coefficient.

SUMMARY OF ORGANIC METHOD CALIBRATIONS

Analysis	Calibration	NPDES [1]		RCRA (SW846) [2]	
		Method	Requirement	Method	Requirement
Aromatic Volatiles by GC	Initial	602	Minimum of 3 levels If % RSD < 10%, use avg RF. Otherwise, calibration curve employed. Six levels for quadratic equation.	8021B	Minimum of 5 levels If % RSD < 20%, use avg RF. Otherwise, calibration curve employed. Six levels for quadratic equation.
	Continuing	602	Analyze QC check sample and evaluate per method requirements	8021B	Mid-level calibration standard analyzed every 10 injections or 12 hrs whichever is greater, % D ≤ 15%, gases 20% D. Evaluate per SOP
	Ending	602	Run closer per NELAC requirement, but no criteria	8021B	Mid-level calibration standard, % D ≤ 15%. Evaluate per SOP, except as noted above.
	Other	602	N/A	8021B	N/A
Herbicides by GC	Initial	615 [9]	Minimum of 3 levels If % RSD < 10%, use avg RF. Otherwise, calibration curve employed	8151A	Minimum of 5 levels If % RSD < 20%, use avg RF. Otherwise, calibration curve employed.
	Continuing	615 [9]	One or more calibration standards analyzed daily % D ± 15% of predicted response	8151A	Mid-level calibration standard analyzed every 10 samples. % D < 15% of predicted response for any analyte quantitated and reported.
	Ending	615 [9]	N/A	8151A	Mid-level calibration standard. % D < 15% of predicted response for any analyte quantitated and reported.
	Other	615 [9]	N/A	8151A	N/A

Analysis	Calibration	NPDES [1]		RCRA (SW846) [2]	
		Method	Requirement	Method	Requirement
Pesticides/ PCBs by GC	Initial	608	Minimum of 3 levels If % RSD < 10%, use avg RF. Otherwise, calibration curve employed	8081A 8082	Minimum of 5 levels. If % RSD < 20%, use avg RF. Otherwise, calibration curve employed. (See SOP NC-GC-038)
	Continuing	608	One or more calibration standards analyzed daily. % D ± 15% of predicted response	8081A 8082	Mid-level calibration standard analyzed every 10 samples. % D < 15% of predicted response for any analyte quantitated and reported.
	Ending	608	N/A	8081A 8082	Mid-level calibration standard. % D < 15% of predicted response for any analyte quantitated and reported.
	Other	608	N/A	8081A 8082	N/A
Petroleum Hydrocarbons/ Oil and Grease	Initial	1664A	Calibrate analytical balance at 2 mg and 1000 mg Calibration must be ± 10% at 2 mg and ± 0.5% at 1000 mg or recalibrate balance	9071B	Calibrate analytical balance at 2 mg and 1000 mg Calibration must be ± 10% at 2 mg and ± 0.5% at 1000 mg or recalibrate balance
	Continuing	1664A	N/A	9071B	N/A
	Ending	1664A	N/A	9071B	N/A

Analysis	Calibration	NPDES [1]		RCRA (SW846) [2]	
		Method	Requirement	Method	Requirement
Purgeable Halocarbons by GC	Initial	601	Minimum of 3 levels If % RSD < 10%, use avg RF. Otherwise, calibration curve employed. Six levels for quadratic equation.	8021B	Minimum of 5 levels If % RSD < 20%, use avg RF. Otherwise, calibration curve employed. Six levels for quadratic equation.
	Continuing	601	Analyze QC check sample and evaluate per method requirements	8021B	Mid-level calibration standard analyzed every 10 injections, or 12 hrs, whichever is greater. % D < 15%, gases 20% D Evaluate per SOP
	Ending	601	Run per NELAC, but no requirement to meet	8021B	Mid-level calibration standard, % D <15%. Evaluate per SOP
	Other	601	N/A	8021B	N/A
Halogenated Volatiles by GC	Initial	--	N/A	8021B	Minimum of 5 levels If % RSD < 20%, use avg RF. Otherwise, calibration curve employed.
	Continuing	--	N/A	8021B	Mid-level calibration standard analyzed every 10 samples. % D < 15% of predicted response for any analyte quantitated and reported.
	Ending	--	N/A	8021B	Mid-level calibration standard % D < 15% of predicted response for any analyte quantitated and reported.
	Other	--	N/A	8021B	N/A

Analysis	Calibration	NPDES [1]		RCRA (SW846) [2]	
		Method	Requirement	Method	Requirement
Semivolatiles	Initial	625	Minimum of 3 levels, lowest near but above MDL. If % RSD ≤ 35%, use avg RF. Otherwise calibration curve employed.	8270C	Minimum of 5 levels, % RSD for RF for CCCs[(4)] < 30% SPCCs[(5)]: RF > 0.050
	Continuing	625	One level every 24 ours. Acceptance criteria are found in the method and SOP.	8270C	Mid-level standard every 12 hours (after tuning) %D for CCCs[(4)] < 20 % between RF from standard and avg RF from initial SPCCs[(5)]: RF > 0.050.
	Ending	625	N/A	8270C	N/A
	Other	625	DFTPP[(7)] tuning every 24 hours before standard or sample runs.	8270C	DFTPP[(7)] tuning at the beginning of every 12 hour shift.

Analysis	Calibration	NPDES [1]		RCRA (SW846) [2]	
		Method	Requirement	Method	Requirement
Volatiles	Initial	624	Minimum of 3 levels, lowest near but above MDL. If % RSD ≤ 35%, use avg RF. Otherwise, calibration curve employed.	8260B	Minimum of 5 levels, %RSD for RF for CCCs[4] < 30.0% SPCCs[5]:RF ≥ 0.300 for Chlorobenzene and 1,1,2,2-tetrachloroethane, Chloromethane and 1,1-dichloroethane, and RF > 0.100 for Bromoform
	Continuing	624	1 level every 24 hours Acceptance criteria are found in the method and SOP	8260B	Mid-level standard every 12 hours (after tuning) %Drift for CCCs(4) < 20.0% between RF from standard and avg RF from initial SPCCs(5): RF ≥ 0.300 for Chlorobenzene and 1,1,2,2-tetrachloroethane, Chloromethane and 1,1-dichloroethane, and RF > 0.100 for Bromoform
	Ending	624	N/A	8260B	N/A
	Other	624	BFB(6)tuning at the beginning of every 24 hour shift.	8260B	BFB(6)tuning at the beginning of every 12 hour shift.

Footnotes:

1 National Pollutant Discharge Elimination System.

2 Resource Conservation and Recovery Act, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, (SW-846), Third Edition, September 1986. Contains Final Update I (July 1992), Final Update IIA (August 1993), Final Update II (September 1994), Final Update IIB (January 1995), and Final Update III (December 1996).

3 TCDD - 2,3,7,8-Tetrachlorodibenzo-p-dioxin.

4 CCC - Continuing Calibration Compounds.

5 SPCC - System Performance Check Compound.

6 BFB – Bromofluorobenzene.

7 DFTPP – Decafluorotriphenylphosphine.

8 Footnote deleted.

9 Method not listed in 40 CFR Part 136.

SECTION 21

MEASUREMENT TRACEABILITY
(NELAC 5.5.6)

21.1 OVERVIEW

Traceability of measurements must be assured using a system of documentation, calibration, and analysis of reference standards. Laboratory equipment that are peripheral to analysis and whose calibration is not necessarily documented in a test method analysis or by analysis of a reference standard must be subject to ongoing certifications of accuracy. At a minimum, these must include procedures for checking specifications of ancillary equipment: balances, thermometers, temperature, Deionized (DI) and Reverse Osmosis (RO) water systems, automatic pipettes and other volumetric measuring devices (refer to Section 20.3). With the exception of Class A Glassware (including glass microliter syringes that have a certificate of accuracy), quarterly accuracy checks are performed for all mechanical volumetric devices. Microsyringes are verified at least semi-annually or disposed of after six months of use. Wherever possible, subsidiary or peripheral equipment is checked against standard equipment or standards that are traceable to national or international standards. Class A glassware should be routinely inspected for chips, acid etching or deformity. If the Class A glassware is suspect, the accuracy of the glassware must be assessed prior to use. Actions to correct or segregate ancillary equipment that does not meet required specifications are identified in the calibration and maintenance section of SOPs and maintenance logbooks for the specific equipment.

21.2 NIST-TRACEABLE WEIGHTS AND THERMOMETERS

Reference standards of measurement must be used for calibration only and for no other purpose, unless it can be shown that their performance as reference standards would not be invalidated.

For NIST-traceable weights and thermometers, the laboratory requires that all calibrations be conducted by a calibration laboratory accredited by A2LA, NVLAP (National Voluntary Laboratory Accreditation Program), APLAC (Asia-Pacific Laboratory Accreditation Cooperation), or EA (European Cooperation for Accreditation). A certificate and scope of accreditation is kept on file at the laboratory.

An external certified service engineer services laboratory balances on an annual basis. This service is documented on each balance with a signed and dated certification sticker. Balance calibrations are checked each day of use. All mercury thermometers are calibrated annually against a traceable reference thermometer. Temperature readings of ovens, refrigerators, and incubators are checked on each day of use.

21.3 REFERENCE STANDARDS / MATERIALS

Reference standards/materials, where commercially available, are traceable to certified reference materials. Commercially prepared standard materials are purchased from vendors accredited by A2LA, NVLAP, with an accompanying Certificate of Analysis that documents the standard purity. If a standard cannot be purchased from a vendor that supplies a Certificate of Analysis, the purity of the standard is documented by analysis. (Refer to Section 9 for additional

information on purchasing). The receipt of all reference standards must be documented. Reference standards are labeled with a unique Standard Identification Number and expiration date. All documentation received with the reference standard is retained as a QC record and references the Standard Identification Number.

All reference, primary and working standards/materials, whether commercially purchased or laboratory prepared, must be checked regularly to ensure that the variability of the standard or material from the 'true' value does not exceed method requirements. The accuracy of calibration standards is checked by comparison with a standard from a second source. In cases where a second standard manufacturer is not available, a vendor-certified different lot is acceptable for use as a second source. For unique situations, where no other source or lot is available, a standard made by a different analyst would be considered a second source. The appropriate Quality Control (QC) criteria for specific standards are defined in laboratory SOPs. In most cases, the analysis of an Initial Calibration Verification (ICV) or LCS (where there is no sample preparation) is used as the second source confirmation. These checks are generally performed as an integral part of the analysis method (e.g., calibration checks, laboratory control samples).

All standards and materials must be stored and handled according to method or manufacturer's requirements in order to prevent contamination or deterioration. Refer to The Corporate Environmental Health & Safety Manual (CW-E-M-001) or laboratory SOPs. For safety requirements, please refer to method SOPs and the laboratory Environmental Health and Safety Manual.

21.4 DOCUMENTATION AND LABELING OF STANDARDS, REAGENTS, AND REFERENCE MATERIALS

Reagents must be at a minimum the purity required in the test method. The date of reagent receipt and the expiration date are documented. The lots for most of the common solvents and acids are tested for acceptability prior to company wide purchase. Refer to TestAmerica's Corporate SOP CA-Q-S-001, Solvent and Acid Lot Testing and Approval.

All manufacturer or vendor-supplied Certificate of Analysis or Purity must be retained, stored appropriately, and readily available for use and inspection. These records are maintained in each group. Records must be kept of the date of receipt and date of expiration of standards, reagents and reference materials. In addition, records of preparation of laboratory standards, reagents, and reference materials must be retained, stored appropriately, and be readily available for use and inspection.

Commercial materials purchased for preparation of calibration solutions, spike solutions, etc., are usually accompanied with an assay certificate or the purity is noted on the label. If the assay purity is 96% or better, the weight provided by the vendor may be used without correction. If the assay purity is less than 96%, a correction must be made to concentrations applied to solutions prepared from the stock commercial material.

21.4.1 All standards, reagents, and reference materials must be labeled in an unambiguous manner. Standards are logged into standard or reagent logbooks, and are assigned a unique identification number. The following information is typically recorded in the Standards Logbook.

- Standard ID

- Description of Standard
- Department
- Preparer's name
- Final volume and number of vials prepared
- Solvent type and lot number
- Preparation date
- Expiration date
- Standard source type (stock or daughter)
- Standard type (spike, surrogate, other)
- Parent standard ID (if applicable)
- Parent standard analyte concentration (if applicable)
- Parent standard amount used (if applicable)
- Component analytes
- Final concentration of each analyte
- Comment box (text field)

Records are maintained in each group for standard and reference material preparation. These records show the traceability to purchased stocks or neat compounds. These records also include method of preparation, date of preparation, expiration date, and preparer's name or initials. Preparation procedures are provided in the Method SOPs.

21.4.2 All standards, reagents, and reference materials must be clearly labeled with a minimum of the following information:

- Expiration date (include prep date for reagents)
- Standard ID (from the Standards Logbook)
- Special health/safety warnings, if applicable

21.4.3 In addition, the following information may be helpful:

- Date of receipt for commercially purchased items or date of preparation for laboratory prepared items
- Date opened (for multi-use containers, if applicable)
- Description of standard (if different from manufacturer's label or if standard was prepared in the laboratory)
- Concentration (if applicable)
- Initials of analyst preparing standard or opening container

All containers of prepared reagents must include a preparation date, expiration date, and an ID number to trace back to preparation.

Procedures for preparation of reagents can be found in the Method SOPs.

Standard ID numbers must be traceable through associated logbooks, worksheets and raw data.

All reagents and standards must be stored in accordance to the following priority:

1) With the manufacturer's recommendations
2) With requirements in the specific analytical methods as specified in the laboratory SOP

SECTION 22

SAMPLING
(NELAC 5.5.7)

22.1 OVERVIEW

The laboratory provides sampling services. Sampling procedures are described in SOP NC-SC-006, Sample Procurement Protocol.

22.2 SAMPLING CONTAINERS

The laboratory offers clean sampling containers for use by clients. These containers are obtained from reputable container manufacturers and meet EPA specifications as required. Any certificates of cleanliness that are provided by the supplier are available from the vendor electronically, or stored at the laboratory.

22.2.1 Preservatives

Upon request, preservatives are provided to the client in pre-cleaned sampling containers. In some cases containers may be purchased pre-preserved from the container supplier. Whether prepared by the laboratory or bought pre-preserved, the grades of the preservatives are at a minimum:

- Hydrochloric Acid – Reagent ACS (Certified VOA Free) or equivalent
- Methanol – Purge and Trap grade
- Nitric Acid – Instra-Analyzed or equivalent
- Sodium Bisulfate – ACS Grade or equivalent
- Sodium Hydroxide – Instra-Analyzed or equivalent
- Sulfuric Acid – Instra-Analyzed or equivalent
- Sodium Thiosulfate – ACS Grade or equivalent

22.3 DEFINITION OF HOLDING TIME

The date and time of sampling documented on the Chain-of-Custody (COC) form establishes the day and time zero. As a general rule, when the maximum allowable holding time is expressed in “days” (e.g., 14 days, 28 days), the holding time is based on calendar day measured. Holding times expressed in “hours” (e.g., 6 hours, 24 hours, etc.) are measured from date and time zero. The first day of holding time ends 24 hours after sampling. Holding times for analysis include any necessary re-analysis. However, there are some programs that determine holding time compliance based on the date and specific time of analysis compared to the time of sampling regardless of holding time length.

22.4 SAMPLING CONTAINERS, PRESERVATION REQUIREMENTS, HOLDING TIMES

The preservation and holding time criteria specified in the following tables are derived from the source documents for the methods. If method-required holding times (refer to Tables 23-1 to 23-7 and in SOPs) or preservation requirements are not met, the reports must be qualified using a flag, footnote, or case narrative. As soon as possible, or "ASAP", is an EPA designation for tests for which rapid analysis is advised; but for which neither EPA nor the laboratory have a basis for a holding time.

22.5 SAMPLE ALIQUOTS / SUBSAMPLING

Taking a representative sub-sample from a container is necessary to ensure that the analytical results are representative of the sample collected in the field. The size of the sample container, the quantity of sample fitted within the container, and the homogeneity of the sample needs consideration when sub-sampling for sample preparation. It is the laboratory's responsibility to take a representative sub-sample or aliquot of the sample provided for analysis. In that regard the following guidelines apply to analysts:

Analysts should handle each sample as if it is potentially dangerous. At a minimum, safety glasses, gloves, and lab coats must be worn when preparing aliquots for analysis.

Guidelines on taking sample aliquots and sub-sampling are located in each analytical SOP.

Tables 23-1 to 23-7 detail holding times, preservation and container requirements, and sample volumes for SDWA and NPDES methods. The sample volumes are intended to be a minimal amount to perform the method. The containers used may be of larger size.

Please note: The holding times are program specific and different programs may have different holding times for equivalent methods, e.g., there are differences in holding times for many organic analytes between SDWA and NPDES. RCRA methods may also be different.

Table 22-1. Inorganic Sample Containers, Preservatives, and Holding Times

Analytical Parameters	Matrix	Minimum Sample Size [1]	NPDES [2, 3, 7]		RCRA (SW846) [3, 4]	
			Method	Requirements	Method	Requirements
Acidity	Water	100 mL	305.1 SM2310B	250 mL plastic or glass. Cool to 4°C, 14 days	---	N/A
Alkalinity, Bicarbonate, Carbonate	Water	100 mL	310.1 2320B	250 mL plastic or glass. Cool to 4°C, 14 days	---	N/A
	Solid	N/A	---	N/A	---	N/A
	Waste	N/A	---	N/A	---	N/A
Ammonia	Water	100 mL	350.1 350.2 SM4500NH_3-E SM4500NH_3-F	500 mL plastic or glass. Cool to 4°CH_2SO_4 to pH < 2, 28 days	---	N/A
	Solid	N/A	---	N/A	---	N/A
	Waste	N/A	---	N/A	---	N/A
Arsenic (ASV) Anodic Stripping Voltammetry	Water	100 mL	---	N/A	7063	250 mL plastic. Cool to 4°C. HCl to pH <2, 28 days
Biochemical Oxygen Demand (BOD), Carbonaceous	Water	1000 mL	405.1 5210B	1000 mL plastic or glass. Cool to 4°C, 48 hours	---	N/A
	Solid	N/A	---	N/A	---	N/A
	Waste	N/A	---	N/A	---	N/A
Bromide	Water	50 mL	300.0A[7]	250 mL plastic or glass. No preservative required, 28 days	9056A	Cool to 4°C. Analyze ASAP after collection
	Solid	N/A	---	N/A	---	N/A
	Waste	N/A	---	N/A	---	N/A
Cation Exchange	Solid	8 oz	---	N/A	9081	8 or 16 oz glass. Cool to 4°C, 6 months
Chemical Oxygen Demand (COD)	Water	100 mL	410.4 5220D	250 mL glass or plastic. Cool to 4°C, H_2SO_4 to pH < 2, 28 days	---	N/A
	Solid	N/A	---	N/A	---	N/A
	Waste	N/A	---	N/A	---	N/A

Analytical Parameters	Matrix	Minimum Sample Size [1]	NPDES [2, 3, 7]		RCRA (SW846) [3, 4]	
			Method	Requirements	Method	Requirements
Chloride	Water	50 mL	300.0A [7] 325.2	250 mL plastic or glass. No preservative required, 28 days	9056A 9251	Method 9056: Cool to 4°C. Analyze ASAP after collection. Method 9251: 250ml plastic or glass, no preservative required, 28 days
	Solid	N/A	---	N/A	---	N/A
	Waste	N/A	---	N/A	---	N/A
Chlorine, Residual	Water	100 mL	330.5	250 mL glass or plastic. Cool to 4°C, analyze immediately	---	N/A
	Solid	N/A	---	N/A	---	N/A
	Waste	N/A	---	N/A	---	N/A
Chromium (Cr^{+6})	Water	100 mL	3500 Cr-D	Method 218.4: 200 mL plastic or glass. Cool to 4°C, 24 hours. Method 3500 Cr-D: 200 mL quartz, TFE, or polypropylene HNO_3 to pH <2. Cool to 4°C. Analyze ASAP after collection	7196A	200 mL plastic or glass. Cool to 4°C, 24 hours
	Solid	20 g	---	N/A	7196A 3060A	250 mL plastic or glass, 30 days to digestion, 96 hours after digestion
	Waste	N/A	---	N/A	---	N/A
Conductivity	Water	100 mL	120.1 2510B	200 mL glass or plastic. Cool to 4°C, 28 days	9050A	200 mL glass or plastic. Cool to 4°C, 24 hours
	Solid	N/A	---	N/A	---	N/A
	Waste	N/A	---	N/A	---	N/A

Analytical Parameters	Matrix	Minimum Sample Size [1]	NPDES [2, 3, 7]		RCRA (SW846) [3, 4]	
			Method	Requirements	Method	Requirements
Cyanide (Amenable)	Water	250 mL	335.1 SM4500CN-G	1 liter plastic or glass, NaOH to pH >12 0.6g ascorbic acid[6]. Cool to 4°C, 14 days unless sulfide is present. Then maximum holding time is 24 hours.	9012A	1 liter plastic or glass, NaOH to pH >12 0.6g ascorbic acid[6]. Cool to 4°C, 14 days
	Solid	50g	---	N/A	9012A	Not Specified
	Waste	50g	---	N/A	9012A	Not Specified
Cyanide (Total)	Water	1L	335.2 335.3 335.4 (7) SM4500CN-E 335.2-CLP-M	1 liter plastic or glass, NaOH to pH >12 0.6g ascorbic acid[6]. Cool to 4°C, 14 days unless sulfide is present. Then maximum holding time is 24 hours.	9012A	1 liter plastic or glass, NaOH to pH >12 0.6g ascorbic acid[6]. Cool to 4°C, 14 days.
	Solid	50g	--	N/A	9012A	8 or 16 oz glass Teflon-lined lids, Cool to 4°C, 14 days
	Waste	50g	--	N/A	9012A	8 or 16 oz glass Teflon-lined lids, Cool to 4°C
Flashpoint (Ignitability)	Liquid	100 mL	---	N/A	1010 ASTM D93-9	No requirements, 250 mL amber glass. Cool to 4°C recommended
	Solid	100 g	--	N/A	---	N/A
	Waste	100 mL	--	N/A	---	N/A
Fluoride	Water	300 mL	300.0 [7] 340.2	500 mL plastic. No preservation required, 28 days.	9056A	Cool to 4°C. Analyze ASAP after collection.
	Solid	N/A	---	N/A	---	N/A
	Waste	N/A	---	N/A	---	N/A

Analytical Parameters	Matrix	Minimum Sample Size [1]	NPDES [2, 3, 7]		RCRA (SW846) [3, 4]	
			Method	Requirements	Method	Requirements
Hardness (Total)	Water	50 mL	130.2 2340B	250 mL glass or plastic, HNO_3 to pH < 2, 6 months	---	N/A
	Solid	N/A	---	N/A	---	N/A
	Waste	N/A	---	N/A	---	N/A
Iron (Ferrous)	Water	100 mL	3500-Fe D	1 liter glass or polyethylene container, 6 months. **This test should be performed in the field.**	-	N/A
	Solid	N/A	-	N/A	-	N/A
	Waste	N/A	-	N/A	-	N/A
Nitrate	Water	50 mL	300.0A [7] SM4500NO_3-E	Method 300.0: 250 mL plastic or glass. Cool to 4°C, 48 hours.	9056A	Method 9056: Cool to 4°C. Analyze ASAP after collection
	Solid	N/A	---	N/A	---	N/A
	Waste	N/A	---	N/A	---	Not Specified
Nitrite	Water	50 mL	300.0A [7] 354.1	250 mL plastic or glass. Cool, 4°C, 48 hours	9056A	Cool, 4°C. Analyze ASAP after collection
	Solid	N/A	---	N/A	---	N/A
	Waste	N/A	---	N/A	---	N/A
Nitrate-Nitrite	Water	50 mL	353.2	250 mL plastic or glass, H_2SO_4 to pH < 2, 28 days	---	N/A
	Solid	N/A	---	N/A	---	N/A
	Waste	N/A	---	N/A	---	N/A
Ortho-phosphate	Water	50 mL	300.0A [7] 365.1 SM4500P-E	100 mL plastic or glass. Filter on site. Cool to 4°C, 48 hours	9056A	Cool to 4°C. Analyze ASAP collection
	Solid	N/A	---	N/A	---	N/A
	Waste	N/A	---	N/A	---	N/A

Analytical Parameters	Matrix	Minimum Sample Size [1]	NPDES [2, 3, 7]		RCRA (SW846) [3, 4]	
			Method	Requirements	Method	Requirements
pH	Water	50 mL	150.1 SM4500H-B	100 mL plastic or glass. Analyze immediately. **This test should be performed in the field.**	9040B	100 mL plastic or glass. Analyze immediately. **This test should be performed in the field.(8)**
	Solid	N/A	---	N/A	9045C	4 oz glass or plastic. Cool to 4°C. Analyze as soon as possible.[8]
	Waste	N/A	---	N/A	9045C	4 oz glass or plastic, Cool to 4°C. Analyze as soon as possible.[8]
Phenolics	Water	100 mL	420.1	500 mL glass, Cool to 4°C, H_2SO_4 to pH < 2, 28 days	9065	1 liter glass recommended, Cool to 4°C, H_2SO_4 to pH < 4, 28 days
	Solid	N/A	---	N/A	---	N/A
	Waste	N/A	---	N/A	9065	Not Specified
Phosphate	Water	50 mL	---	N/A	9056A	Cool to 4°C, analyze ASAP collection
	Solid	N/A	---	N/A	9056A	N/A
	Waste	N/A	---	N/A	9056A	N/A

Analytical Parameters	Matrix	Minimum Sample Size [1]	NPDES [2, 3, 7]		RCRA (SW846) [3, 4]	
			Method	Requirements	Method	Requirements
Phosphorus (Total)	Water	100 mL	365.2 365.1 SM4500P-E	100 mL plastic or glass, H_2SO_4 to pH < 2, 28 days	---	N/A
	Solid	N/A	---	N/A	---	N/A
	Waste	N/A	---	N/A	---	N/A
Settleable Solids	Water	1000 mL	160.5	1000 mL plastic or glass. Cool to 4°C, 48 hours	---	N/A
	Solid	N/A	---	N/A	---	N/A
	Waste	N/A	---	N/A	---	N/A
Specific Conductance	Water	50 mL	120.1 2510B	250 mL plastic or glass. Cool to 4°C, 24 hours	9050A	250 mL plastic or glass. Cool to 4°C, 28 days
	Solid	N/A	---	N/A	---	N/A
	Waste	N/A	---	N/A	---	N/A
Sulfate (SO_4)	Water	50 mL	300.0A [7] 375.4	100 mL plastic or glass. Cool to 4°C, 28 days	9056A 9038	Method 9056: Cool to 4°C. Analyze ASAP collection. Method 9038: 200 mL plastic or glass, Cool to 4°C, 28 days
	Solid	N/A	---	N/A	---	N/A
	Waste	100 mL	---	N/A	9038	200 mL plastic or glass. Cool to 4°C, 28 days

Analytical Parameters	Matrix	Minimum Sample Size [1]	NPDES [2, 3, 7]		RCRA (SW846) [3, 4]	
			Method	Requirements	Method	Requirements
Sulfide	Water	250 mL	376.1	500 mL plastic or glass. Cool to 4°C, Add 2 mL zinc acetate plus NaOH to pH > 9, 7 days	9030A 9030B/ 9034	500 mL plastic, No headspace. Cool to 4°C. Add 4 drops of 2N zinc acetate per 100 mL of sample, adjust the pH to > 9 with 6 N NaOH solution, 7 days
	Solid	50 g	---	N/A	9030A 9030B/ 9034	Cool to 4°C. Fill surface of solid with 2N Zinc acetate until moistened. Store headspace-free
	Waste	50 g	---	N/A	9030A 9030B/ 9034	Cool to 4°C. Fill surface of solid with 2N Zinc acetate until moistened. Store headspace-free
Total Dissolved Solids (Filterable)	Water	100 mL	160.1 2540C	250 mL plastic or glass. Cool to 4°C, 7 days	---	N/A
	Solid	N/A	---	N/A	---	N/A
	Waste	N/A	---	N/A	---	N/A

Analytical Parameters	Matrix	Minimum Sample Size [1]	NPDES [2, 3, 7]		RCRA (SW846) [3, 4]	
			Method	Requirements	Method	Requirements
Total Kjeldahl Nitrogen (TKN)	Water	100 mL	351.3	500 mL plastic or glass. Cool to 4°C, H_2SO_4 to pH < 2, 28 days	---	N/A
	Solid	N/A	---	N/A	---	N/A
	Waste	N/A	---	N/A	---	N/A
Total Organic Carbon (TOC)	Water	100 mL	415.1 SM5310D	100 mL plastic or glass. Cool to 4°C, H_2SO_4 to pH < 2, 28 days	9060 Walkley-Black	100 mL glass or 40 mL VOA vials,Cool to 4°C, H_2SO_4 or HCl to pH < 2, 28 days
	Solid	N/A	---	N/A	9060 Walkley-Black	Not Specified
	Waste	N/A	---	N/A	9060 Walkley-Black	Not Specified
Total Organic Halides (TOX) (EOX)	Water	100 mL	450.1 (7)	500 mL amber glass, Teflon®-lined lid. Cool to 4°C, HNO_3 to pH <2, no headspace, 28 days	9020B 9023 (EOX)	500 mL amber glass, Teflon®-lined lid. Cool to 4°C, H_2SO_4 to pH < 2, no headspace, 28 days
	Solid	N/A	---	N/A	---	N/A
	Waste	N/A	---	N/A	---	N/A
Total Solids	Water	100 mL	160.3	250 mL plastic or glass. Cool to 4°C, 7 days	---	N/A
	Solid	N/A	---	N/A	---	N/A
	Waste	N/A	---	N/A	---	N/A
Total Suspended Solids (Nonfilterable)	Water	100 mL	160.2	250 mL plastic or glass. Cool, 4°C, 7 days	---	N/A
	Solid	N/A	---	N/A	---	N/A
	Waste	N/A	---	N/A	---	N/A

Analytical Parameters	Matrix	Minimum Sample Size [1]	NPDES [2, 3, 7]		RCRA (SW846) [3, 4]	
			Method	Requirements	Method	Requirements
Turbidity	Water	50 mL	180.1	250 mL plastic or glass. Cool, 4°C, 48 hours	---	N/A
	Solid	N/A	---	N/A	---	N/A
	Waste	N/A	---	N/A	---	N/A
Volatile Solids	Water	100 mL	160.4	250 mL plastic or glass. Cool, 4°C, 7 days	---	N/A
	Solid	N/A	---	N/A	---	N/A
	Waste	N/A	---	N/A	---	N/A
Metals (excludes Hg)	Water	100 mL	200 series	1 liter glass or polyethylene container, HNO_3 to pH $\leq$ 2, 6 months	6010B 6020	1 liter glass or polyethylene container, HNO_3 to pH $\leq$ 2, 6 months
	Solid	200 g	200 series	2, 8, or 16 oz glass or polyethylene container storage at 4 °C	6010B 6020	8 or 16 oz glass or polyethylene container, storage at 4°C, 6 months
	Waste	200 g	200 series	N/A	6010B 6020	8 or 16 oz glass or polyethylene container, storage at 4°C, 6 months
Mercury (CVAA) (CVAFS)	Water	100 mL	245.1 1631E	250 mL glass or polyethylene container, HNO_3 to pH $\leq$ 2, 28 days	7470A	1 liter glass or polyethylene container, HNO_3 to pH $\leq$ 2, 28 days
	Solid	200 g	--	2, 8, or 16 oz glass or polyethylene container. Cool to 4°C, 28 days. Not applicable for Method 1631E.	7471A	8 or 16 oz glass or polyethylene container. Cool to 4°C, 28 days (CORP-MT-0007)
	Waste	200 g	--	N/A	7471A	8 or 16 oz glass or polyethylene container. Cool, 4°C, 28 days (CORP-MT-0007)

Footnotes

[1] Minimum sample size indicates sample amount needed for a single analysis. Matrix spikes or duplicates will require an additional sample amount of at least this amount for each additional QC sample aliquot required.

[2] National Pollutant Discharge Elimination System - MCAWW, March 1983.

[3] Holding times are calculated from date of collection. Holding Times are determined based on date of collection to preparation/analysis.

[4] Resource Conservation and Recovery Act, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, (SW-846), Third Edition, September 1986. Contains Final Update I (July 1992), Final Update IIA, (August 1993), Final Update II (September 1994), Final Update IIB (January 1995), and Final Update III (December 1996).

[5] Solid matrix type includes soil, sediment, sludge and other solid materials not classified as waste.

[6] Samples to be analyzed for cyanide should be field-tested for residual chlorine. If residual chlorine is detected, ascorbic acid should be added.

[7] Method not listed in 40 CFR Part 136.

[8] If not done in the field (ASAP) per the method and requested by client, analyze in lab within 48 hours.

[9] EPA issued memo recommending not to use reactive cyanide and sulfide methods.

Table 22-2. Organic Sample Containers, Preservatives, and Holding Times

Analytical Parameters	Matrix	Minimum Sample Size [1]	NPDES [2, 3]		RCRA (SW846) [3, 4]	
			Method	Requirements	Method [6]	Requirements
Aromatic Volatiles	Water	40 mL	602	40 mL glass, VOA vial (in triplicate) with Teflon®-lined septa without headspace. Cool to 4°C, 7 days with pH > 2,14 days with pH ≤ 2	8021B	40 mL glass, VOA vial (in triplicate) with Teflon®-lined septa without headspace. Cool to 4°C, 1:1 HCl to pH ≤2, 14 days with pH ≤ 2
	Solid [5]	5 g or 25 g	--	N/A	8021B	4 or 8 oz glass with Teflon®-lined lid. Cool to 4 °C, 14 days. Field preserved with methanol for medium level analysis. Soil sample can also be taken by using the EnCore™ sampler and preserved in the lab within 48 hours of sampling. Maximum holding time for Encore Sampler is 48 hours (before the sample is added to methanol). Cool, 4°C [12]
	Waste	5 g or 25 g	--	N/A	8021B	4 or 8 oz glass with Teflon®-lined lid. Cool to 4 °C, 14 days. Field preserved with methanol for medium level analysis. Soil sample can also be taken by using the EnCore™ sampler and preserved in the lab within 48 hrs of sampling. Max holding time for EnCore™ sampler is 48 hrs (before the sample is added to methanol). Cool to 4°C [12]

Analytical Parameters	Matrix	Minimum Sample Size [1]	NPDES [2, 3]		RCRA (SW846) [3, 4]	
			Method	Requirements	Method [6]	Requirements
Halogenated Volatiles by GC	Water	40 mL	--	N/A	8021B	40 mL glass, VOA vial (in triplicate) with Teflon®-lined septa without headspace. Cool to 4°C, 1:1 HCl to pH ≤ 2, 14 days
	Solid [5]	5 g or 25 g	--		8021B	4 or 8 oz glass with Teflon®-lined lid. Cool to 4 °C, 14 days. Field preserved with methanol for medium level analysis. Soil sample can also be taken by using the EnCore™ sampler and preserved in the lab within 48 hours of sampling. Maximum holding time for Encore Sampler is 48 hours (before the sample is added to methanol). Cool to 4°C [12]
	Waste	5 g or 25 g	--	N/A	8021B	4 or 8 oz glass with Teflon®-lined lid. Cool to 4 °C, 14 days. Field preserved with methanol for medium level analysis. Soil sample can also be taken by using the EnCore™ sampler and preserved in the lab within 48 hours of sampling. Maximum holding time for Encore Sampler is 48 hours (before the sample is added to methanol). Cool, 4°C [12].

Analytical Parameters	Matrix	Minimum Sample Size [1]	NPDES [2, 3]		RCRA (SW846) [3, 4]	
			Method	Requirements	Method [6]	Requirements
Herbicides	Water	1L	615 [(10)]	1 liter amber glass with Teflon®-lined lid, Sodium thiosulfate or ascorbic acid if residual chlorine present. Cool to 4°C, Extraction, 7 days. Analysis, 40 days after extraction	8151A	1 liter amber glass with Teflon®-lined lid. If residual chlorine present, add 3 mL sodium thiosulfate per gallon. Cool to 4°C. Extraction, 7 days. Analysis, 40 days of the start of extraction.
	Solid	50 g	--	N/A	8151A	4 or 8 oz glass widemouth with Teflon®-lined lid. Cool to 4 °C. Extraction, 14 days. Analysis, 40 days of the start of the extraction.
	Waste	50 g	--	N/A	8151A	4 or 8 oz glass widemouth with Teflon®-lined lid. Cool to 4 °C. Extraction, 14 days. Analysis, 40 days of the start of the extraction.

Analytical Parameters	Matrix	Minimum Sample Size [1]	NPDES [2, 3]		RCRA (SW846) [3, 4]	
			Method	Requirements	Method [6]	Requirements
Pesticides/ PCBs	Water	1L	608	1 liter amber glass with Teflon®-lined lid, Adjust pH to 5-9 if extraction not to be done within 72 hours of sampling. Add sodium thiosulfate if residual chlorine present and aldrin is being determined. Cool, 4°C. Extraction, 7 days. Analysis, 40 days after extraction.	8081A 8082	1 liter amber glass with Teflon®-lined lid, If residual chlorine present, add 3 mL 10% sodium thiosulfate per gallon. Cool, 4°C. Extraction, 7 days. Analysis, 40 days of the start of the extraction.
	Solid	50 g	---	N/A	8081A 8082	4 or 8 oz glass wide mouth with Teflon®-lined lid. Cool, 4°C. Extraction, 14 days. Analysis, 40 days of the start of the extraction.
	Waste	50 g	---	N/A	8081A 8082	4 or 8 oz glass wide mouth with Teflon®-lined lid. Cool, 4°C. Extraction, 14 days. Analysis, 40 days of the start of the extraction.

Analytical Parameters	Matrix	Minimum Sample Size [1]	NPDES [2, 3]		RCRA (SW846) [3, 4]	
			Method	Requirements	Method [6]	Requirements
Petroleum Hydrocarbons /Oil and Grease	Water	1 L	1664A[(7)]	1 liter glass, Cool, 4°C HCl or H_2SO_4 to pH <2 28 days	9071B	1 liter glass, Cool, 0-4°C HCl or H_2SO_4 to pH <2 28 days
	Solid	30 g	1664A[(7)]	8 or 16 oz. Wide mouth glass jar, Cool, 4°C, 28 days	9071B	8 or 16 oz. wide mouth glass jar, Cool, 0-4°C, 28 days
	Waste	---	---	N/A	9071B	N/A
Purgeable Halocarbons by GC	Water	40 mL	601	40 mL glass VOA vial (in triplicate) with Teflon®-lined septa with no headspace, Cool, 4°C, 14 days.	8021B	40 mL glass VOA vial (in triplicate) with Teflon®-lined septa with no headspace, Cool, 4°C, 1:1 HCl to pH $\leq$ 2, 14 days.
	Solid	5 g or 25 g	---	N/A	8021B	4 or 8 oz glass with Teflon®-lined lid, Cool 4 °C, 14 days. Field preserved with methanol for medium level analysis. Soil sample can also be taken by using the EnCore™ sampler and preserved in the lab within 48 hours of sampling. Maximum holding time for EnCore™ sampler is 48 hrs (before the sample is added to methanol). Cool, 4°C[12].

Analytical Parameters	Matrix	Minimum Sample Size [1]	NPDES [2, 3]		RCRA (SW846) [3, 4]	
			Method	Requirements	Method [6]	Requirements
Purgeable Halocarbons by GC (cont'd)	Waste	5 g or 25 g	---	N/A	8021B	4 or 8 oz glass with Teflon®-lined lid, Cool 4 °C, 14 days. Field preserved with methanol for medium level analysis. Soil sample can also be taken by using the EnCore™ sampler and preserved in the lab within 48 hours of sampling. Maximum holding time for Encore™ sampler is 48 hours (before the sample is added to methanol). Cool, 4°C[12]
Semivolatiles	Water	1L	625	1 liter amber glass with Teflon®-lined lid. Cool, 4°C. Extraction, 7 days. Analysis, 40 days.	8270C	1 liter amber glass with Teflon®-lined lid, If residual chlorine present, add 3 mL sodium thiosulfate per gallon. Cool, 4°C. Extraction, 7 days. Analysis, within 40 days of extraction.
	Solid	50 g	---	N/A	8270C	8 or 16 oz glass wide mouth with Teflon-lined lid. Cool, 4°C. Extraction, 14 days. Analysis, within 40 days of extraction.
	Waste	50 g	---	N/A	8270C	8 or 16 oz glass wide mouth with Teflon®-lined lid. Cool, 4°C. Extraction, 14 days. Analysis, within 40 days of extraction.

Analytical Parameters	Matrix	Minimum Sample Size [1]	NPDES [2, 3]		RCRA (SW846) [3, 4]	
			Method	Requirements	Method [6]	Requirements
Volatile Organics	Water	40 mL	624	40 mL glass, VOA vial (in triplicate) with Teflon®-lined septa without headspace. Cool to 4°C. Add sodium thiosulfate if residual chlorine, 7 days with pH > 2, 14 days with pH ≤ 2[8].	8260B	40 mL glass, VOA vial (in triplicate) with Teflon®-lined septa without headspace. Cool to 4°C. Add sodium thiosulfate if residual chlorine, 1:1 HCl to pH ≤ 2, 14 days with pH ≤ 2[9].
	Solid[5]	5 g or 25 g	--	N/A	8260B	4 or 8 oz glass with Teflon®-lined lid. Cool to 4 °C, 14 days. Field preserved with sodium bisulfate solution for low level analysis, or with methanol for medium level analysis. Soil sample can also be taken by using the EnCore™ sampler and preserved in the lab within 48 hrs of sampling. Maximum holding time for EnCore™ sampler is 48 hrs (before the sample is added to methanol or sodium bisulfate). Cool to 4°C[(12)]
	Waste	5 g or 25 g	--	N/A	8260B	4 or 8 oz glass with Teflon®-lined lid, Cool 4 °C, 14 days. Field preserved with sodium bisulfate solution for low level analysis, or with methanol for medium level analysis. Soil sample can also be taken by using the EnCore™ sampler and preserved in the lab within 48 hrs of sampling. Maximum holding time for Encore™ sampler is 48 hrs (before sample is added to methanol or sodium bisulfate). Cool to 4°C[12]

Footnotes

[1] Minimum sample size indicates sample amount needed for a single analysis. Matrix spikes or duplicates will require an additional sample amount of at least this amount for each additional QC sample aliquot required.

[2] National Pollutant Discharge Elimination System - 40 CFR Part 136, Appendix A.

[3] Holding times are calculated from the date of collection.

[4] Resource Conservation and Recovery Act, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, Third Edition, September 1986. Contains Final Update I (July 1992), Final Update IIA (August 1993), Final Update II (September 1994), Final Update IIB (January 1995), and Final Update III (December 1996).

[5] Solid matrix type includes soil, sediment, sludge or other solids not classified as waste.

[6] Only one determination method is listed when separate methods are required for preparation and analysis.

[7] Method 1664 was promulgated by the EPA with an effective date of June 14, 1999.

[8] For acrolein and acrylonitrile the pH should be adjusted to 4-5. This pH adjustment is not required if acrolein is not measured. Samples requiring analysis of acrolein that received no pH adjustment must be analyzed within three days of sampling.

[9] For acrolein and acrylonitrile the pH should be adjusted to 4-5.

[10] Method not listed in 40 CFR Part 136.

[11] Should only be used in the presence of residual chlorine.

[12] Depending on regulatory programs, EnCore™ samplers may be preserved for up to 14 days from sampling by freezing at -5 to-12°C until analysis. Alternatively the EnCore™ sample may be transferred to a 40-ml VOA vial and preserved by freezing at -5 to -12°C until analysis. Some regulatory agencies may require 4 or 8 oz glass with Teflon®-lined lid, Cool 4°C, 14 days. This technique is not recommended, but will be supported where required. (Preservation and holding times are subject to client specifications.)

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Table 22-3. Sample Containers, Preservatives, and Holding Times for TCLP[1] and SPLP[2]

Analytical Parameters	Matrix	Minimum Sample Size	TCLP Method 1311 and SPLP Method 1312 Requirements	
			From Field Collection to TCLP/SPLP Extraction	From TCLP/SPLP Extraction to Analysis
Mercury	Liquid Solid Waste	1L	1L glass, Cool, 4°C, 28 days	Glass or polyethylene 28 days
Metals (except mercury)	Liquid Solid Waste	1L	1L glass, Cool, 4°C, 180 days	Glass or polyethylene 180 days
Semivolatiles	Liquid Solid Waste	1L	1L glass, Cool 4°C, 14 days	1L glass Extraction of leachate within 7 days of TCLP extraction, Analyze extract within 40 days
Volatiles	Liquid Solid Waste	6 oz	4 oz glass, Cool 4°C, 14 days	40 mL glass, 14 days

Footnotes

[1] TCLP = Toxicity Characteristic Leaching Procedure
[2] SPLP = Synthetic Precipitation Leaching Procedure

SECTION 23

HANDLING OF SAMPLES
(NELAC 5.5.8)

Sample management procedures at the laboratory ensure that sample integrity and custody are maintained and documented from sampling/receipt through disposal.

23.1 CHAIN OF CUSTODY (COC)

The COC form is the written documented history of any sample and is initiated when bottles are sent to the field, or at the time of sampling. This form is completed by the Sampling personnel and accompanies the samples to the laboratory where it is received and stored under the laboratory's custody. The purpose of the COC form is to provide a legal written record of the handling of samples from the time of collection until they are received at the laboratory. It also serves as the primary written request for analyses from the client to the laboratory. The COC form acts as a purchase order for analytical services when no other contractual agreement is in effect. An example of a COC form may be found in Figure 23-1.

23.1.1 Field Documentation

The information the sampler needs to provide at the time of sampling on the container label is:

- Sample identification
- Date and time
- Preservative

During the sampling process, the COC form is completed and must be legible (see Figure 23-1). This form includes information such as:

- Client name, address, phone number and fax number (if available)
- Project name and/or number
- The sample identification
- Date, time and location of sampling
- Sample collectors name
- The matrix description
- The container description
- The total number of each type of container
- Preservatives used
- Analysis requested
- Requested turnaround time (TAT)
- Any special instructions
- Purchase Order number or billing information (e.g. quote number) if available
- The date and time that each person received or relinquished the sample(s), including their signed name.

The samples are stored in a cooler with ice, as applicable, and remain solely in the possession of the client's field technician until the samples are delivered to the laboratory. The sample collector must assure each container is in his/her physical possession or in his/her view at all times, or stored in such a place and manner to preclude tampering. The field technician relinquishes the samples in writing on the COC form to the Sample Control personnel at the laboratory or to a TestAmerica courier. Samples are only considered to be received by lab when personnel at the laboratory have physical contact with the samples.

Note: Independent couriers are not required to sign the COC form. The COC is usually kept in the sealed sample cooler. The COC is stored with project information and the report.

23.1.2 Legal / Evidentiary Chain-of-Custody

The lab does not accept samples that require legal Chain-of-Custody.

23.2 SAMPLE RECEIPT

Samples are received at the laboratory by designated Sample Receiving personnel, and a unique laboratory project identification number is assigned. Each sample container must be assigned a unique sample identification number that is cross-referenced to the client identification number such that traceability of test samples is unambiguous and documented. Each sample container is affixed with a durable sample identification label. Sample acceptance, receipt, tracking, and storage procedures are summarized in the following sections. SOP NC-SC-005, Sample Receiving and Sample Control, describes the laboratory's sample receipt procedure.

23.2.1 Laboratory Receipt

Samples must be received and logged in at TestAmerica by a designated sample custodian or other properly trained associate. Upon sample receipt, the sample custodian shall, as appropriate:

- Wear appropriate personal protective equipment. At a minimum, this consists of cut-resistant gloves, a lab coat, and safety glasses
- Examine the shipping containers to verify that the custody tape is intact
- Examine all sample containers for damage
- Open shipping containers in adequately ventilated areas to assure worker safety
- Determine if the temperature required by the requested testing program has been maintained during shipment. Document the shipping container temperature on the Cooler Receipt Form
- Compare samples received against those listed on the COC
- Verify that sample holding times have not been exceeded
- Examine all shipping records for accuracy and completeness
- Determine sample pH (if required for the scheduled analysis) (except VOA and TOX samples) and record on the Cooler Receipt Form (CRF)
- Sign and date the COC immediately (only after shipment is accepted) and attach the waybill
- Note any problems associated with the coolers and samples on the cooler receipt form and

notify the PM who in turn notifies the client

- Attach durable (water-resistant) laboratory sample container labels with unique laboratory identification number and test
- Place the samples in proper laboratory storage.

A Cooler Receipt Form (CRF) or an equivalent form/system is generated by sample control during the sample log-in process to document anomalies identified upon the receipt of samples in the laboratory. These anomalies are outside of laboratory control and do not require corrective actions to be taken within the laboratory. The affected client must be notified by the PM or designee of all CRFs generated for their samples. The PM is responsible for resolving with the client how to proceed with the samples and documenting the decision to proceed with the analysis of compromised samples. CRFs must be resolved prior to sample preparation and analysis. The completed CRF must be stored in the project file. An example CRF is shown in Figure 24-4. The report narrative must include an explanation of sample receiving related anomalies.

23.2.1.1 Unique Sample Identification

A unique lot number is generated for each project that is logged. The lot number is nine characters in length, and is based on the date of receipt. For example, Lot **A9C010121** is described as follows:

A - TestAmerica location where the samples were received (A = North Canton, C = Pittsburgh, etc.)

9 - Last digit of the year (i.e., 2009)

C - Month (i.e., A = January, B = February, C = March)

01 - These two numeric characters identify the day of the month--in this case, the first day of the month.

0121 - These four numeric characters are the sequential assignment of numbers specific to each lot received. Each day, the first lot logged receives the number "0101"; the second lot receives the number "0102", etc.

As each sample is logged, individual *sample numbers* are created in sequential order, noted as the three-digit suffix following the lot number. For example, four samples were received on May 10, 2008 for PCB analysis with the following client IDs: AB100, AB101, AB102, AB103.

Client ID - Lot No. - Sample No.	Analysis
AB100A8E100101-001	PCB
AB101A8E100101-002	PCB
AB102A8E100101-003	PCB
AB103A8E100101-004	PCB

If a client requests an MS/MSD for a sample(s), it is denoted in LIMS by an "S" for the MS and a "D" for the MSD.

Work Order Numbers: As each parameter is logged, a random unique 8-digit Work Order number is generated by LIMS. For example, Work Order **A5WE1-1-1C** is described as follows:

A5WE1 - The first five characters of the Work Order number identifies each unique sample.

1 - The "modifier" indicates the run number. This number will always be a "1" at log-in. However, additional modifier numbers may be generated by the analytical lab groups when necessary (example: for a re-extract).

1C - The "suffix" identifies a specific test for each sample, but is unique to the test being performed on each sample.

Each sample container representing the same sample (as indicated by the client ID) will have the same 5-digit Work Order number, along with a 3-digit suffix that corresponds to the bottle count (example: -001, -002, -003, etc.). For example, if five containers are submitted for the same sample and the LIMS-generated Work Order number is CREE4, LIMS will generate five labels--CREE4-001, CREE4-002, CREE4-003, CREE4-004, and CREE4-005. These numbers are used to track sample containers through the analytical process.

23.3 Sample Acceptance Policy

The laboratory has a written sample acceptance policy outlined in SOP NC-SC-005, Sample Receiving and Sample Control, that clearly outlines the circumstances under which samples must be accepted or rejected. These include:

- A COC filled out completely
- Samples must be properly labeled
- Proper sample containers with adequate volume for the analysis and necessary QC
- Samples must be preserved according to the requirements of the requested analytical method
- Sample holding times must be adhered to
- All samples submitted for water/solid Volatile Organic analyses must have a Trip Blank submitted at the same time
- The Project Manager must be notified if any sample is received in damaged condition.

Data from samples which do not meet these criteria are flagged and the nature of the variation from policy is defined. A copy of the sample acceptance policy is provided to each client prior to shipment of samples.

Once sample acceptance is verified, the samples are logged into LIMS according to SOP NC-SC-005.

23.4 SAMPLE STORAGE

In order to avoid deterioration, contamination or damage to a sample during storage and handling, from the time of receipt until all analyses are complete, samples are stored in refrigerators suitable for the sample matrix. Metals samples may be unrefrigerated. In addition, samples to be analyzed for volatile organic parameters are stored in separate refrigerators designated for volatile organic parameters only. Samples are never to be stored with reagents, standards, or materials that may create contamination.

To ensure the integrity of the samples during storage, refrigerator blanks are maintained in the volatile sample refrigerators and analyzed every week.

Access to the laboratory is controlled such that sample storage need not be locked at all times unless a project specifically demands it. Samples are accessible to laboratory personnel only. Visitors to the laboratory are prohibited from entering the refrigerator and laboratory areas unless accompanied by an employee of TestAmerica.

23.5 HAZARDOUS SAMPLES AND FOREIGN SOILS

All samples per SOP are treated as hazardous. If any extra or known hazards are present in the sample, the sample is flagged and precautions / instructions are put in the comments. Hazardous samples are segregated out, and go into the waste stream profile for the nature of the hazard. All soils--foreign and domestic--go to a USDA approved incinerator.

23.6 SAMPLE SHIPPING

In the event the laboratory needs to ship samples, the samples are placed in a cooler with enough ice to ensure the samples remain just above freezing and at or below 6.0°C during transit. The samples are carefully surrounded by packing material to avoid breakage (yet maintain appropriate temperature). A trip blank is enclosed for those samples requiring water/solid volatile organic analyses. The Chain-of-Custody form is signed by the Sample Control technician and attached to the shipping paperwork. Samples are generally shipped overnight express or hand-delivered by a TestAmerica courier to maintain sample integrity. All personnel involved with shipping and receiving samples must be trained to maintain the proper Chain-of-Custody documentation and to keep the samples intact and on ice. The Environmental, Health and Safety Manual contains additional shipping requirements.

Note: If a client does not request trip blank analysis on the COC or other paperwork, the laboratory will not analyze the trip blanks that were supplied. However, in the interest of good client service, the laboratory will advise the client at the time of sample receipt that it was noted that they did not request analysis of the trip blank; and that the laboratory is providing the notification to verify that they are not inadvertently omitting a key part of regulatory compliance testing.

23.7 SAMPLE DISPOSAL

Samples should be retained for a minimum of 30 days after the project report is sent; however, provisions may be made for earlier disposal of samples once the holding time is exceeded. Some samples are required to be held for longer periods based on regulatory or client

requirements (e.g., 60 days after project report is sent). The laboratory must follow the longer sample retention requirements where required by regulation or client agreement. Several possibilities for sample disposal exist--the sample may be consumed completely during analysis, the sample may be returned to the customer or location of sampling for disposal, or the sample may be disposed of in accordance with the laboratory's waste disposal procedures (SOP NC-SC-005, Sample Receiving and Sample Control). All procedures in the laboratory Environmental, Health and Safety Manual are followed during disposal. Samples are normally maintained in the laboratory no longer than two months from receipt unless otherwise requested. Unused portions of samples found or suspected to be hazardous according to state or federal guidelines may be returned to the client upon completion of the analytical work. Waste disposal complies with all federal and state laws and regulations.

If a sample is part of a known litigation, the affected legal authority, sample data user, and/or submitter of the sample must participate in the decision about the sample disposal. All documentation and correspondence concerning the disposal decision process must be kept on file. Pertinent information includes the date of disposal, nature of disposal (such as sample depletion, hazardous waste facility disposal, return to client), and names of individuals who conducted the arrangements and physically completed the task. Sample labels are destroyed through the disposal method, e.g., samples are incinerated. A Waste Manifest is completed.

Figure 23-1. Example: Chain of Custody (COC)

TestAmerica North Canton
4101 Shuffle Drive N.W.

North Canton, OH 44720
phone 330-497-9396 fax 330-497-0772

Chain of Custody Record

Client Contact	**Project Manager:**	**Site Contact:**	**Date:**
Your Company Name here	**Tel/Fax:**	**Lab Contact:**	**Carrier:**
Address	**Analysis Turnaround Time**		
City/State/Zip	Calendar (C) or Work Days (W)		
(xxx) xxx-xxxx Phone	TAT if different from Below _____		
(xxx) xxx-xxxx FAX	☐ 2 weeks		
Project Name:	☐ 1 week		
Site:	☐ 2 days		
P O #	☐ 1 day		

Sample Identification	**Sample Date**	**Sample Time**	**Sample Type**	**Matrix**	**# of Cont.**	**Filtered Sample**

Preservation Used: 1= Ice, 2= HCl; 3= H2SO4; 4=HNO3; 5=NaOH; 6= Other _____

Possible Hazard Identification

☐ *Non-Hazard* ☐ *Flammable* ☐ *Skin Irritant* ☐ *Poison B* ☐ *Unknown*

Sample Disposal (A fee may be assessed if samples are retained longer than 1 month)

☐ *Return To Client* ☐ *Disposal By Lab* ☐ *Archive For _____ Months*

Special Instructions/QC Requirements & Comments:

Relinquished by:	Company:	Date/Time:	Received by:	Company:
Relinquished by:	Company:	Date/Time:	Received by:	Company:
Relinquished by:	Company:	Date/Time:	Received by:	Company:

Figure 23-2.

Example: Custody Seal

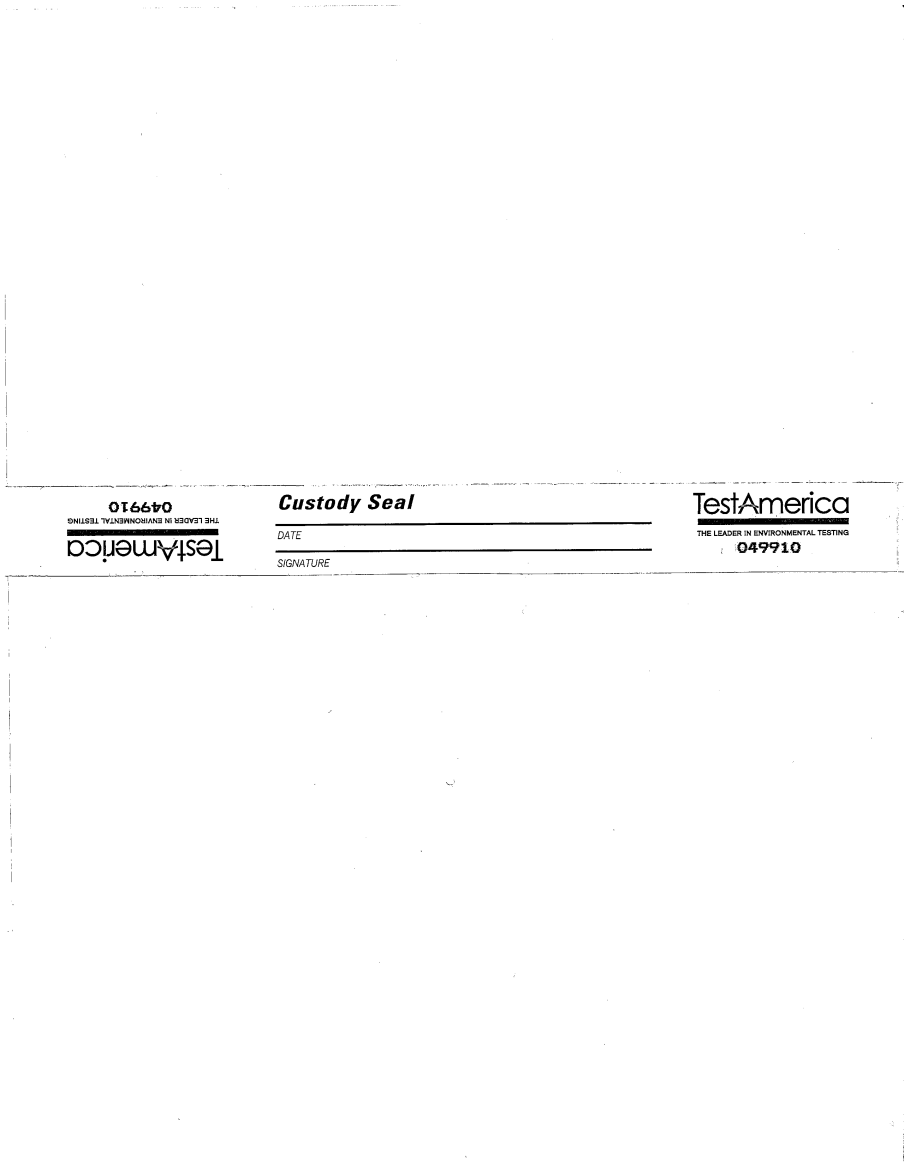


Figure 23-3. Example: Internal Chain of Custody (COC)

TestAmerica Laboratories, Inc.
Sample Control Record

Client:

Lot Number:

Case Number/SDG:

Storage Location:

Laboratory Sample ID	Transferred By	Date	Entered	Removed	Reason	Date Returned

Figure 23-4. Example: Cooler Receipt Form (Page 1)

TestAmerica Cooler Receipt Form/Narrative **Lot Number:**_____
North Canton Facility

Client _____ Project _____ By _____

Cooler Received on _____ Opened on _____ (Signature)
FedEx ☐ UPS ☐ DHL ☐ FAS ☐ Stetson ☐ Client Drop Off ☐ TestAmerica Courier ☐ Other _____

TestAmerica Cooler # _____ Multiple Coolers ☐ Foam Box ☐ Client Cooler ☐ Other_____

1. Were custody seals on the outside of the cooler(s)? Yes ☐ No ☐ Intact? Yes ☐ No ☐ NA ☐
   If YES, Quantity_____ Quantity Unsalvageable _____
   Were custody seals on the outside of cooler(s) signed and dated? Yes ☐ No ☐ NA ☐
   Were custody seals on the bottle(s)? Yes ☐ No ☐
   If YES, are there any exceptions? _____
2. Shipper's packing slip attached to the cooler(s)? Yes ☐ No ☐
3. Did custody papers accompany the sample(s)? Yes ☐ No ☐ Relinquished by client? Yes ☐ No ☐
4. Were the custody papers in the appropriate place? Yes ☐ No ☐
5. Packing material used: Bubble Wrap ☐ Foam ☐ None ☐ Other _____
6. Cooler temperature upon receipt _____ °C See back of form for multiple coolers/temps ☐

METHOD: IR ☐ Other ☐ _____
COOLANT: Wet Ice ☐ Blue Ice ☐ Dry Ice ☐ Water ☐ None ☐

7. Did all bottles arrive in good condition (Unbroken)? Yes ☐ No ☐
8. Could all bottle labels be reconciled with the COC? Yes ☐ No ☐
9. Were sample(s) at the correct pH upon receipt? Yes ☐ No ☐ NA ☐
10. Were correct bottle(s) used for the test(s) indicated? Yes ☐ No ☐
11. Were air bubbles >6 mm in any VOA vials? Yes ☐ No ☐ NA ☐
12. Sufficient quantity received to perform indicated analyses? Yes ☐ No ☐
13. Was a Trip Blank present in the cooler(s)? Yes ☐ No ☐ Were VOAs on the COC? Yes ☐ No ☐

Contacted PM _____ Date _____ by _____ via Verbal ☐ Voice Mail ☐ Other ☐
Concerning_____

14. CHAIN OF CUSTODY

The following discrepancies occurred:
_____
_____
_____
_____
_____
_____

15. SAMPLE CONDITION

Sample(s)_____ were received after the recommended holding time had expired.
Sample(s)_____ were received in a broken container.
Sample(s)_____were received with bubble >6 mm in diameter. (Notify PM).

16. SAMPLE PRESERVATION

Sample(s) _____ were further preserved in Sample Receiving to meet recommended pH level(s). *Nitric Acid Lot* # 100108-HNO_3 - *Sulfuric Acid Lot* # 100108-H_2SO_4; *Sodium Hydroxide Lot* # 073007-*NaOH; Hydrochloric Acid Lot* # 092006-*HCl; Sodium Hydroxide and Zinc Acetate Lot* # 050205-$(CH_3COO)_2ZN$/*NaOH.* What time was preservative added to sample(s)? _____

Client ID	pH	Date	Initials

Example: Cooler Receipt Form (Page 2)

TestAmerica Cooler Receipt Form/Narrative
North Canton Facility

Client ID	pH	Date	Initials

Cooler	Temp °C	Method	Coolant

Discrepancies Cont'd:

SECTION 24

ASSURING THE QUALITY OF TEST RESULTS
(NELAC 5.5.9)

24.1 OVERVIEW

In order to assure our clients of the validity of their data, the laboratory continuously evaluates the quality of the analytical process. The analytical process is controlled not only by instrument calibration as discussed in Section 20, but also by routine process quality control measurements (e.g., Blanks, Laboratory Control Samples (LCS), Matrix Spikes (MS), duplicates (DUP), surrogates, Internal Standards (IS)). These quality control checks are performed as required by the method or regulations to assess precision and accuracy. In addition to the routine process quality control samples, Proficiency Testing (PT) Samples (concentrations unknown to laboratory) are analyzed to help ensure laboratory performance.

24.2 CONTROLS

Sample preparation or pre-treatment is commonly required before analysis. Typical preparation steps include homogenization, grinding, solvent extraction, sonication, acid digestion, distillation, reflux, evaporation, drying and ashing. During these pre-treatment steps, samples are arranged into discreet manageable groups referred to as preparation (prep) batches. Prep batches provide a means to control variability in sample treatment. Control samples are added to each prep batch to monitor method performance and are processed through the entire analytical procedure with investigative/field samples.

24.3 NEGATIVE CONTROLS

Table 24-1. Example – Negative Controls

Control Type	Details
Method Blanks (MB)	are used to assess preparation and analysis for possible contamination during the preparation and processing steps.
	The specific frequency of use for method blanks during the analytical sequence is defined in the specific standard operating procedure for each analysis. Generally it is 1 for each batch of samples; not to exceed 20 environmental samples.
	The method blank is prepared from a clean matrix similar to that of the associated samples that is free from target analytes (e.g., Reagent water, Ottawa sand, glass beads, etc.) and is processed along with and under the same conditions as the associated samples. The method blank goes through all of the steps of the process (including as necessary: filtration, clean-ups, etc.).
Calibration Blanks	are prepared and analyzed along with calibration standards where applicable or injected at specifed frequencies throughout an analytical sequence. They are prepared using the same reagents that are used to prepare the standards. In some analyses the calibration blank may be included in the calibration curve. These blanks may be termed Initial Calibration Blanks (ICB) or Continuing Calibration Blanks (CCB),

Table 24-1. Example – Negative Controls

Control Type	Details
Instrument Blanks	are blank reagents or reagent water that may be processed during an analytical sequence in order to assess contamination in the analytical system. In general, instrument blanks are used to differentiate between contamination caused by the analytical system and that caused by the sample handling or sample prep process. Instrument blanks may also be inserted throughout the analytical sequence to minimize the effect of carryover from samples with high analyte content.
Trip Blanks [1]	are required to be submitted by the client with each shipment of samples requiring aqueous and solid volatiles analyses. Additionally, trip blanks may be prepared and analyzed for volatile analysis of air samples, when required by the client. A trip blank may be purchased (certified clean) or is prepared by the laboratory by filling a clean container with pure deionized water that has been purged to remove any volatile compounds. Appropriate preservatives are also added to the container. The trip blank is sent with the bottle order and is intended to reflect the environment that the containers are subjected to throughout shipping and handling and help identify possible sources if contamination is found. The field sampler returns the trip blank in the cooler with the field samples.
Field Blanks [1]	are sometimes used for specific projects by the field samplers. A field blank prepared in the field by filling a clean container with pure reagent water and appropriate preservative, if any, for the specific sampling activity being undertaken. (EPA OSWER)
Equipment Blanks [1]	are also sometimes created in the field for specific projects. An equipment blank is a sample of analyte-free media which has been used to rinse common sampling equipment to check effectiveness of decontamination procedures. (NELAC)
Holding Blanks	also referred to as refrigerator or freezer blanks, are used to monitor the sample storage units for volatile organic compounds during the storage of VOA samples in the laboratory

[1] When known, these field QC samples should not be selected for matrix QC as it does not provide information on the behavior of the target compounds in the field samples. Usually, the client sample ID will provide information to identify the field blanks with labels such as "FB", "EB", or "TB."

Evaluation criteria and corrective action for these controls are defined in the specific standard operating procedure for each analysis.

24.4 POSITIVE CONTROLS

Control samples (e.g., QC indicators) are analyzed with each batch of samples to evaluate data based upon:

1) Method Performance [Laboratory Control Sample (LCS) or Blank Spike (BS)], which entails both the preparation and measurement steps

2) Matrix Effects (Matrix Spike (MS) or Sample Duplicate (MD, DUP), which evaluates field sampling accuracy, precision, representativeness, interferences, and the effect of the matrix on the method performed.

Each regulatory program and each method within those programs specify the control samples that are prepared and/or analyzed with a specific batch.

Note that frequency of control samples vary with specific regulatory, methodology, and project-specific criteria. Complete details on method control samples are as listed in each analytical SOP.

24.4.1 Method Performance Control - Laboratory Control Sample (LCS)

24.4.1.1 The LCS measures the accuracy of the method in a blank matrix and assesses method performance independent of potential field sample matrix affects in a laboratory batch.

24.4.1.2 The LCS is prepared from a clean matrix similar to that of the associated samples that is free from target analytes (for example: Reagent water, Ottawa sand, glass beads, etc.) and is processed along with and under the same conditions as the associated samples. The LCS is spiked with verified known amounts of analytes or is made of a material containing known and verified amounts of analytes, taken through all preparation and analysis steps along with the field samples. Where there is no preparation taken for an analysis (such as in aqueous volatiles), or when all samples and standards undergo the same preparation and analysis process (such as Phosphorus), a calibration verification standard is reported as the LCS. In some instances where there is no practical clean solid matrix available, aqueous LCS's may be processed for solid matrices; final results may be calculated as mg/kg or ug/kg, assuming 100% solids and a weight equivalent to the aliquot used for the corresponding field samples, to facilitate comparison with the field samples.

24.4.1.3 Certified pre-made reference material purchased from a NIST/A2LA accredited vendor may also be used for the LCS when the material represents the sample matrix or the analyte is not easily spiked (e.g., solid matrix LCS for metals, TDS, etc.).

24.4.1.4 The specific frequency of use for LCS during the analytical sequence is defined in the specific standard operating procedure for each analysis. It is generally one for each batch of sample--not to exceed 20 environmental samples.

24.4.1.5 If the mandated or requested test method, or project requirements, do not specify the spiking components, the laboratory shall spike all reportable components to be reported in the Laboratory Control Sample (and Matrix Spike) where applicable, e.g., no spike of pH. However, in cases where the components interfere with accurate assessment (such as simultaneously spiking chlordane, toxaphene and PCBs in Method 608), the test method has an extremely long list of components or components are incompatible, at a minimum, a representative number of the listed components (see below) shall be used to control the test method. The selected components of each spiking mix shall represent all chemistries, elution patterns and masses, permit specified analytes and other client requested components. However, the laboratory shall ensure that all reported components are used in the spike mixture within a two-year time period.

24.4.1.5.1 For methods that have 1-10 target analytes, spike all components.

24.4.1.5.2 For methods that include 11-20 target analytes, spike at least 10 or 80%, whichever is greater.

24.4.1.5.3 For methods with more than 20 target analytes, spike at least 16 components.

24.4.1.5.4 Exception: Due to analyte incompatibility in pesticides, Toxaphene and Chlordane are only spiked at client request based on specific project needs.

24.4.1.5.5 Exception: Due to analyte incompatibility between the various PCB Aroclors, Aroclors 1016 and 1260 are used for spiking as they cover the range of all of the Aroclors. Specific Aroclors may be used by request on a project-specific basis.

24.5 SAMPLE MATRIX CONTROLS

Table 24-2 Sample Matrix Control

Control Type	Details	
Matrix Spikes (MS)	Use	To assess the effect sample matrix of the spiked sample has on the precision and accuracy of the results generated by the method used;
	Typical Frequency [1]	At a minimum, with each matrix-specific batch of samples processed, an MS is carried through the complete analytical procedure. Unless specified by the client, samples used for spiking are randomly selected and rotated between different client projects.If the mandated or requested test method does not specify the spiking components, the laboratory shall spike all reportable components to be reported in the Laboratory Control Sample and Matrix Spike. Refer to the method SOP for complete details
	Description	Essentially, a sample fortified with a known amount of the test analyte(s).
Surrogate	Use	Measures method performance to sample matrix (organics only).
	Typical Frequency [1]	Are added to all samples, standards, and blanks, for all organic chromatography methods except when the matrix precludes its use or when a surrogate is not available. The recovery of the surrogates is compared to the acceptance limits for the specific method. Poor surrogate recovery may indicate a problem with sample composition and shall be reported, with data qualifiers, to the client whose sample produced poor recovery.
	Description	Are similar to matrix spikes except the analytes are compounds with properties that mimic the analyte of interest and are unlikely to be found in environment samples.
Duplicates[2]	Use	For a measure of analytical precision, with each matrix-specific batch of samples processed, a matrix duplicate (MD or DUP) sample, matrix spike duplicate (MSD), or LCS duplicate (LCSD) is carried through the complete analytical procedure.
	Typical Frequency [1]	Duplicate samples are usually analyzed with methods that do not require matrix spike analysis.
	Description	Performed by analyzing two aliquots of the same field sample independently or an additional LCS.
Internal Standards	Use	Are spiked into all environmental and quality control samples (including the initial calibration standards) to monitor the qualitative aspect of organic and some inorganic analytical measurements.
	Typical Frequency [1]	All organic and ICP methods as required by the analytical method.
	Description	Used to correct for matrix effects and to help troubleshoot variability in analytical response and are assessed after data acquisition. Possible sources of poor internal standard response are sample matrix, poor analytical technique or instrument performance.

[1] See the specific analytical SOP for type and frequency of sample matrix control samples.
[2] LCSD's are normally not performed except when regulatory agencies or client specifications require them. The recoveries for the spiked duplicate samples must meet the same laboratory established recovery limits as the accuracy QC samples. If an LCSD is analyzed both the LCS and LCSD must meet the same recovery criteria and be included in the final report. The precision measurement is reported as "Relative Percent Difference" (RPD). Poor precision between duplicates (except LCS/LCSD) may indicate non-homogeneous matrix or sampling.

24.6 ACCEPTANCE CRITERIA (CONTROL LIMITS)

24.6.1 As mandated by the test method and regulation, each individual analyte in the LCS, MS, or Surrogate Spike is evaluated against the control limits published in the test method. Where there are no established acceptance criteria, the laboratory calculates in-house control limits with the use of control charts or, in some cases, utilizes client project-specific control

limits. When this occurs, the regulatory or project limits will supersede the laboratory's in-house limits.

Note: For methods, analytes, and matrices with very limited data (e.g., unusual matrices not analyzed often), interim limits are established using available data or by analogy to similar methods or matrices.

Note: For Ohio VAP the laboratory must implement Corrective Action procedures to resolve the deviation and limit qualification of the final results. The laboratory is not permitted to deviate from its VAP approved SOP if it intends to attest under affidavit that the "results" are VAP certified. When all corrective actions listed in the SOP have been exhausted, it may be necessary to use technical judgment in which case the decision process and rationale will be presented in the final report and/or affidavit and the data will be noted as 'not VAP certified' on the affidavit.

24.6.2 Once control limits have been established, they are verified, reviewed, and updated if necessary on an annual basis unless the method requires more frequent updating. Control limits are established per method (as opposed to per instrument) regardless of the number of instruments utilized.

24.6.3 Laboratory-generated Percent Recovery acceptance (control) limits are generally established by taking $\pm$ 3 Standard Deviations (99% confidence level) from the average recovery of a minimum of 20-30 data points (more points are preferred).

24.6.3.1 Regardless of the calculated limit, the limit should be no tighter than the Calibration Verification (ICV/CCV), (unless the analytical method specifies a tighter limit).

24.6.3.2 In-house limits cannot be any wider than those mandated in a regulated analytical method. Client or contract required control limits are evlauted against the laboratory's statistically derived control limits to determine if the data quality objectives (DQOs) an be achieved. If laboratory control limits are not consistent with DQOs, then alternatives must be considered, such as method improvements or use of an alternate analytical method.

24.6.3.3 The lowest acceptable recovery limit will be 10% (the analyte must be detectable and identifiable). Exception: The lowest acceptable recovery limit for Benzidine will be 5%, and the analyte must be detectable and identifiable.

24.6.3.4 The maximum acceptable recovery limit will be 200%.

24.6.3.5 The maximum acceptable RPD limit will be 30% for organic methods and 20% for inorganic methods. The minimum RPD limit is 10%.

24.6.3.6 If either the high or low end of the control limit changes by $\leq$ 10% from previous, the control chart is visually inspected and, using professional judgment, they may be left unchanged if there is no affect on laboratory ability to meet the existing limits.

24.6.4 The lab must be able to generate a current listing of their control limits and track when the updates are performed. In addition, the laboratory must be able to recreate historical

control limits. Refer to NC-QA-018, Statistical Evaluation of Data and Development of Control Charts, for details.

24.6.4.1 One example: The QA Department generates a Quality Control Limit Summary that contains tables that summarize the precision and accuracy acceptability limits for analyses. Unless otherwise noted, limits within these tables are laboratory generated. Once reviewed and approved, the limits are entered into LIMS and are effective immediately. The Quality Assurance department maintains an archive of all limits used within the laboratory.

24.6.5 An LCS that is within the acceptance criteria establishes that the analytical system is in control and is used to validate the process. Samples that are analyzed with an LCS with recoveries outside of the acceptance limits may be determined as out of control and should be re-analyzed if possible. If re-analysis is not possible, then the results for all affected analytes for samples within the same batch must be qualified when reported. The internal Corrective Action process (see Section 12) is also initiated if an LCS exceeds the acceptance limits. Sample results may be qualified and reported without re-analysis if:

24.6.5.1 The analyte results are below the reporting limit and the LCS is above the upper control limit.

24.6.5.2 If the analytical results are above the relevant regulatory limit and the LCS is below the lower control limit.

Note: For Ohio VAP the laboratory must implement Corrective Action procedures to resolve the deviation and limit qualification of the final results. The laboratory is not permitted to deviate from its VAP approved SOP if it intends to attest under affidavit that the "results" are VAP certified. When all corrective actions listed in the SOP have been exhausted, it may be necessary to use technical judgment in which case the decision process and rationale will be presented in the final report and/or affidavit and the data will be noted as ‘not VAP certified’ on the affidavit.

24.6.5.3 Or, for NELAC and Department Of Defense (DOD) work, there are an allowable number of Marginal Exceedances (ME):

- <11 analytes 0 marginal exceedances are allowed.
- 11 – 30 Analytes 1 marginal exceedance is allowed
- 31-50 Analytes 2 marginal exceedances are allowed
- 51-70 Analytes 3 marginal exceedances are allowed
- 71-90 Analytes 4 marginal exceedances are allowed
- > 90 Analytes 5 marginal exceedances are allowed

24.6.5.3.1 Marginal exceedances are recovery exceedances between 3 SD and 4 SD from the mean recovery limit (NELAC).

Note: Use of Marginal Exceedances is not permitted for Ohio VAP.

24.6.5.3.2 Marginal exceedances must be random. If the same analyte exceeds the LCS control limit repeatedly, it is an indication of a systematic problem. The source of the error must be located and corrective action taken. The laboratory has a system to monitor marginal exceedances to ensure that they are random.

24.6.5.3.3 Though marginal excedences may be allowed, the data must still be qualified to indicate it is outside of the normal limits.

24.6.6 If the MS/MSDs do not meet acceptance limits, the MS/MSD and the associated spiked sample is reported with a qualifier for those analytes that do not meet limits. If obvious preparation errors are suspected, or if requested by the client, unacceptable MS/MSDs are reprocessed and re-analyzed to prove matrix interference. A more detailed discussion of acceptance criteria and corrective action can be found in the lab's method SOPs and in Section 12.

24.6.7 If a surrogate standard falls outside the acceptance limits, and if there is not obvious chromatographic matrix interference, re-analyze the sample to confirm a possible matrix effect. If the recoveries confirm or there was obvious chromatographic interference, results are reported from the original analysis and a qualifier is added. If the re-analysis meets surrogate recovery criteria, the second run is reported (or both are reported if requested by the client).

Note: A more detailed discussion of acceptance criteria and corrective action can be found in the laboratory's method SOPs and in Section 12.

24.7 ADDITIONAL PROCEDURES TO ASSURE QUALITY CONTROL

24.7.1 The laboratory has written and approved method SOPs to assure the accuracy of the test method including calibration (see Section 20), use of certified reference materials (see Section 21), and use of PT samples (see Section 15).

24.7.2 A discussion regarding MDLs, Limit of Detection (LOD), and Limit of Quantitation (LOQ) can be found in Section 19.

24.7.3 Use of formulae to reduce data is discussed in the method SOPs and in Section 20.

24.7.4 Selection of appropriate reagents and standards is included in Sections 9 and 21.

24.7.5 A discussion on selectivity of the test is included in Section 5.

24.7.6 Constant and consistent test conditions are discussed in Section 18.

24.7.7 The laboratory sample acceptance policy is included in Section 23.

SECTION 25

REPORTING RESULTS
(NELAC 5.5.10)

25.1 OVERVIEW

The results of each test are reported accurately, clearly, unambiguously, and objectively in accordance with State and Federal regulations as well as client requirements. Analytical results are issued in a format that is intended to satisfy customer and laboratory accreditation requirements as well as provide the end user with the information needed to properly evaluate the results. Where there is a conflict between client requests and laboratory ethics or regulatory requirements, the laboratory's ethical and legal requirements are paramount, and the laboratory must work with the client during project setup to develop an acceptable solution. Refer to Section 7.

A variety of report formats are available to meet specific needs.

In cases where a client asks for simplified reports, there must be a written request from the client. There still must be enough information that would show any analyses that were out of conformance (QC out of limits) and there should be a reference to a full report that is made available to the client.

Review of reported data is included in Section 19.

25.2 TEST REPORTS

Analytical results are reported in a format that is satisfactory to the client and meets all requirements of applicable accrediting authorities and agencies. A variety of report formats are available to meet specific needs. The report is printed, reviewed, and signed by the appropriate Project Manager. At a minimum, the standard laboratory report shall contain the following information:

25.2.1 A report title with a "Sample Result" header.

25.2.2 Each report cover page printed, which includes the laboratory name, address, and telephone number.

25.2.3 A unique identification of the report (e.g., Work Order number) and on each page an identification in order to ensure the page is recognized as part of the report and a clear identification of the end.

Note: Page numbers of report are represented at the bottom of each page. The report is sequentially paginated. The final page of the report is labeled as "End of Report".

25.2.4 A copy of the Chain-of-Custody (COC).

- Any COCs involved with subcontracting are included.

- Any additional addenda to the report must be treated in a similar fashion so it is a recognizable part of the report and cannot accidentally get separated from the report (e.g., Sampling information).

25.2.5 The name and address of client and a project name/number, if applicable.

25.2.6 Client project manager or other contact

25.2.7 Description and unambiguous identification of the tested sample(s) including the client identification code.

25.2.8 Date of receipt of sample, date and time of collection, and date(s) of test preparation and performance, and time of preparation or analysis.

25.2.9 Date reported or date of revision, if applicable

25.2.10 Method of analysis including method code (EPA, Standard Methods, etc)

25.2.11 Reporting limit

25.2.12 Method detection limits (if requested)

25.2.13 Definition of data qualifiers and reporting acronyms, e.g., ND

25.2.14 Sample results

25.2.15 QC data consisting of method blank, surrogate, LCS, and MS/MSD recoveries and control limits

25.2.16 Condition of samples at receipt including temperature. This may be accomplished in a narrative or by attaching sample login sheets (refer to Section 25.2.4 – Item 3, regarding additional addenda).

25.2.17 A statement to the effect that the results relate only to the items tested and the sample as received by the laboratory.

25.2.18 A signature and title of the person(s) accepting responsibility for the content of the report and date of issue. Signatories are appointed by the Lab Director.

25.2.19 When NELAC accreditation is required, the lab must certify that the test results meet all requirements of NELAC or provide reasons and/or justification if they do not.

25.2.20 The laboratory includes a cover page.

25.2.21 Where applicable, a narrative to the report that explains the issue(s) and corrective action(s) taken in the event that a specific accreditation or certification requirement was not met.

25.2.22 When soil samples are analyzed, a specific identification as to whether soils are reported on a “wet weight” or “dry weight” basis.

25.2.23 Appropriate laboratory certification number for the state of origin of the sample, if applicable.

25.2.24 If only part of the report is provided to the client (client requests some results before all of it is complete), it must be clearly indicated on the report, e.g., partial report, or how your lab identifies it. A complete report must be sent once all of the work has been completed.

25.2.25 Any non-TestAmerica subcontracted analysis results are provided as a separate report on the official letterhead of the subcontractor. All TestAmerica subcontracting is clearly identified on the report as to which laboratory performed a specific analysis.

Note: Refer to the Corporate SOP on Electronic Reporting and Signature Policy CA-L-P-002 for details on internally applying electronic signatures of approval.

25.2.26 Reports for Ohio VAP work require a VAP affidavit be completed and included with the report.

Note: For additional information on Ohio VAP affidavits refer to OAC Rule 3745-300-04 and OAC Rule 3745-300-13(N), effective March 1, 2009.

25.3 REPORTING LEVEL OR REPORT TYPE

The laboratory offers two levels of quality control reporting. Each level, in addition to its own specific requirements, contains all the information provided in the preceding level. The packages provide the following information in addition to the information described above:

- Standard report – all features in Section 25.2
- Expanded deliverable – standard report presented on CLP-like forms and relevant calibration information. All supporting raw data is supplied.

In addition to the various levels of QC packaging, the laboratory also provides reports in diskette deliverable form. Procedures used to ensure client confidentiality are outlined in Section 25.7.

25.3.1 Electronic Data Deliverables (EDDs)

EDDs are routinely offered as part of TestAmerica services. TestAmerica North Canton offers a variety of EDD formats including (but not limited to) ADR, EQuIS, GISKey, Region 5, NJHAZsite, and a wide variety of client specific multi-file, Excel and flat file formats.

EDD specifications are submitted to the IT Department by the PM for review and undergo the contract review process. Once the facility has committed to providing data in a specific electronic format, the coding of the format may need to be performed. This coding is documented and validated. The validation of the code is retained by the IT staff coding the EDD.

EDDs must be subject to a review to ensure their accuracy and completeness. If EDD generation is automated, review may be reduced to periodic screening if the laboratory can demonstrate that it can routinely generate that EDD without errors. Any revisions to the EDD format must be reviewed until it is demonstrated that it can routinely be generated without errors. If the EDD can be reproduced accurately and if all subsequent EDDs can be produced error-free, each EDD does not necessarily require a review.

25.4 SUPPLEMENTAL INFORMATION FOR TEST

The lab identifies any unacceptable QC analyses or any other unusual circumstances or observations such as environmental conditions and any non-standard conditions that may have affected the quality of a result. This is typically in the form of a footnote or a qualifier and/or a narrative explaining the discrepancy in the front of the report.

25.4.1 Numeric results with values outside of the calibration range, either high or low are qualified as 'estimated'.

25.4.2 Where quality system requirements are not met, a statement of compliance/non-compliance with requirements and/or specifications is required, including identification of test results derived from any sample that did not meet NELAC sample acceptance requirements such as improper container, holding time, or temperature.

25.4.3 Where applicable, a statement on the estimated uncertainty of measurements; information on uncertainty is needed when a client's instructions so require.

25.4.4 Opinions and Interpretations - The test report contains objective information, and generally does not contain subjective information such as opinions and interpretations. If such information is required by the client, the Laboratory Director will determine if a response can be prepared. If so, the Laboratory Director will designate the appropriate member of the management team to prepare a response. The response must be fully documented, and reviewed by the Laboratory Director, before release to the client. There may be additional fees charged to the client at this time, as this is a non-routine function of the laboratory.

When opinions or interpretations are included in the report, the laboratory provides an explanation as to the basis upon which the opinions and interpretations have been made. Opinions and interpretations are clearly noted as such and where applicable, a comment should be added suggesting that the client verify the opinion or interpretation with their regulator.

25.5 ENVIRONMENTAL TESTING OBTAINED FROM SUBCONTRACTORS

If the laboratory is not able to provide the client the requested analysis, the samples would be subcontracted following the procedures outlined in the Corporate SOP CA-L-S-002, Subcontracting.

Data reported from analyses performed by a subcontractor laboratory are clearly identified as such on the analytical report provided to the client. Results from a subcontract laboratory outside of the TestAmerica network are reported to the client on the subcontract laboratory's original report stationary and the report includes any accompanying documentation.

25.6 CLIENT CONFIDENTIALITY

In situations involving the transmission of environmental test results by telephone, facsimile, or other electronic means, client confidentiality must be maintained.

TestAmerica will not intentionally divulge to any person (other than the client or any other person designated by the client in writing) any information regarding the services provided by TestAmerica or any information disclosed to TestAmerica by the client. Furthermore, information known to be potentially endangering to national security or an entity's proprietary rights will not be released.

Note: This shall not apply to the extent that the information is required to be disclosed by TestAmerica under the compulsion of legal process. TestAmerica will, to the extent feasible, provide reasonable notice to the client before disclosing the information.

Note: Authorized representatives of an accrediting authority are permitted to make copies of any analyses or records relevant to the accreditation process, and copies may be removed from the laboratory for purposes of assessment.

25.6.1 Report deliverable formats are discussed with each new client. If a client requests that reports be faxed or e-mailed, the reports are faxed with a cover sheet or e-mailed with the following note that includes a confidentiality statement similar to the following:

"Confidentiality Notice: The information contained in this message is intended only for the use of the addressee, and may be confidential and/or privileged. If the reader of this message is not the intended recipient, or the employee or agent responsible to deliver it to the intended recipient, you are hereby notified that any dissemination, distribution or copying of this communication is strictly prohibited. If you have received this communication in error, please notify the sender immediately."

25.7 FORMAT OF REPORTS

The format of reports is designed to accommodate each type of environmental test carried out and to minimize the possibility of misunderstanding or misuse.

25.8 AMENDMENTS TO TEST REPORTS

Corrections, additions, or deletions to reports are only made when justification arises through supplemental documentation. Justification is documented using the laboratory's corrective action system (refer to Section 12).

When the report is re-issued, a notation of "report reissue" is placed on the cover/signature page of the report or at the top of the narrative page with a brief explanation of reason for the reissue and a reference back to the lst final report generated.

25.9 POLICIES ON CLIENT REQUESTS FOR AMENDMENTS

25.9.1 Policy on Data Omissions or Reporting Limit Increases

Fundamentally, our policy is simply to not omit previously reported results (including data qualifiers) or to not raise reporting limits and report sample results as ND. This policy has few exceptions. Exceptions are:

- Laboratory error
- Sample identification is indeterminate (confusion between COC and sample labels).
- An incorrect analysis (not analyte) was requested (e.g., COC lists 8315 but client wanted 8310). A written request for the change is required.
- Incorrect limits reported based on regulatory requirements
- The requested change has absolutely *no possible* impact on the interpretation of the analytical results and there is *no possibility* of the change being interpreted as misrepresentation by anyone inside or outside of our company.

25.9.2 Multiple Reports

TestAmerica does not issue multiple reports for the same work order where there is different information on each report (this does not refer to copies of the same report) unless required to meet regulatory needs and approved by QA.

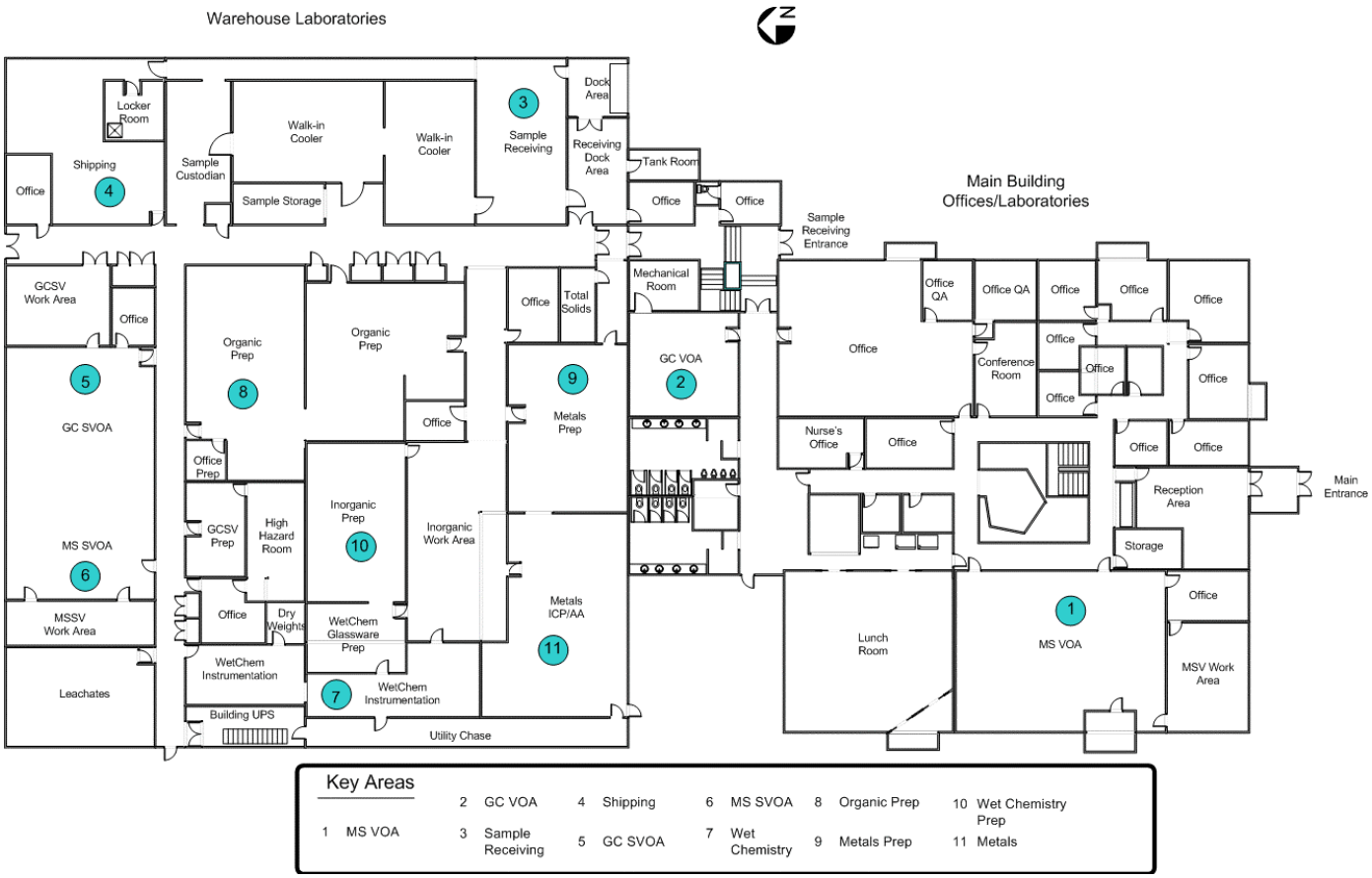
Appendix 1.

Laboratory Floor Plan

TestAmerica – North Canton

4101 Shuffel Dr NW
North Canton, OH 44720

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING



Appendix 2. Laboratory Method Listing

Wet Chemistry Methods [1]

Analytical Parameters	Matrix		Fields of Testing		
			CWA	RCRA (SW846)	Other
Acidity	Water		305. [2] SM 2310 B	--	--
Alkalinity, Bicarbonate, Carbonate	Water		305. [2] SM 2320 B		
	Solid		EPA 310.1[2] (M)	--	--
Arsenic (ASV) Anodic Stripping Voltammetry	Water		--	EPA 7063	--
Ash Content	Solid		--	--	ASTM D29-74
Biochemical Oxygen Demand, Carbonaceous	Water		EPA 405.1 SM 5210 B	--	--
Bromide	Water		EPA 300.0A	EPA 9056A	--
	Waste		EPA 300.0A	EPA 9056A	--
	Solid		EPA 300.0A (M)	EPA 9056A	--
Cation-Exchange Capacity	Solid		--	EPA 9081	--
Chemical Oxygen Demand	Water		EPA 410.4 SM 5220D	--	--
	Waste		EPA 410.4	--	--
Chloride	Water		EPA 300.0A EPA 325.2[2]	EPA 9056A EPA 9251	EPA 325.2[2]
	Waste		EPA 300.0A	EPA 9056A	--
	Solid		EPA 300.0A (M)	EPA 9056A EPA 9251(M)	--
Chromium, Hexavalent	Water		EPA 3500-Cr-D	EPA 7196A	--
	Waste		EPA 3500-Cr-D	EPA 7196A	--
	Solid		--	EPA 3060A EPA 7196A	--

[1] Any matrix not listed is not applicable for the associated method
[2] Removed from 40CFR

Analytical Parameters	Matrix	Fields of Testing		
		CWA	RCRA (SW846)	Other
Specific Conductance	Water	EPA 120.1 SM 2510B	EPA 9050A	--
	Waste	EPA 120.1	EPA 9050A	--
	Solid	--	EPA 9050A	--
Chlorine, Residual	Water	EPA 330.5[2] SM 3500 CL-G	--	--
Cyanide (Amenable)	Water	EPA 335.1[2] SM 4500 CN-G	EPA 9012A	--
	Solid	--	EPA 9012A	--
Cyanide (Total)	Water	SM 4500-CN E EPA 335.4 335.2-CLP-M	EPA 9012A	---
	Waste	--	EPA 9012A	--
	Solid	--	EPA 9012A	--
Cyanide (Weak and Dissociable) (Free)	Water	SM 4500-CN I	--	--
Dissolved Oxygen	Water	360.1[2] SM 4500 O-G	--	--
Flash Point	Waste	--	EPA 1010	ASTM D93-9
	Solid	--	EPA 1010	ASTM D93-9
Fluoride	Water	EPA 300.0A EPA 340.2[2]	EPA 9056A	SM 4500 F-C, ISE
	Waste	EPA 340.2 (M)[2] EPA 300.0A (M)	EPA 9056A	--
	Solid	EPA 340.2 (M)[2] EPA 300.0A (M)	EPA 9056A	--
Iron, Ferrous & Ferric	Water	SM 3500 FE D	--	--
Hardness	Water	EPA 130.2[2]	--	SM 2340B
Moisture	Solid	---	EPA 160.3 (M) ASTM D2216-90	---
Nitrogen, Ammonia	Water	EPA 350.1	--	EPA 350.2[2]
	Waste	EPA 350.1	--	EPA 350.2[2]
	Solid	EPA 350.1	--	EPA 350.2[2]
	Water	SM 4500 NH_3-B (Distillation)	--	--
	Water	SM 4500 NH_3-E (Titration)	--	--
	Water	SM 4500 NH_3-F (ISE)	--	--

Analytical Parameters	Matrix		Fields of Testing		
			CWA	RCRA (SW846)	Other
Nitrite (NO_2)	Water		EPA 300.0A	EPA 9056A	--
	Waste		EPA 300.0A (M)	EPA 9056A	--
	Solid		EPA 300.0A (M)	EPA 9056A	--
Nitrate (NO_3)	Water		EPA 300.0A	EPA 9056A	SM 4500 NO_3-E
	Waste		EPA 300.0A (M)	EPA 9056A	--
	Solid		EPA 300.0A (M)	--	--
Nitrate plus Nitrite NO_2/NO_3	Water		EPA 353.2	--	--
	Waste		EPA 353.2	--	--
Total Kjeldahl Nitrogen (TKN)	Water		EPA 351.3	--	SM 4500 NO_3
	Waste		EPA 351.3	--	--
	Solid		EPA 351.3	--	--
Oil and Grease (Hexane Extractable Material)	Water		EPA 1664A	EPA 9071B	--
	Waste		EPA 1664A	EPA 9071B	--
	Solid		--	EPA 9071B	--
Ortho-phosphate o-PO_4	Water		EPA 300.0A EPA 365.1	EPA 9056A	SM 4500 P-E
	Waste		EPA 300.0A (M)	EPA 9056A	--
	Solid		EPA 300.0A (M) EPA 365.1	EPA 9056A	--
pH	Water		EPA 150.1[2]	EPA 9040B	EPA 9041
	Waste		SM 4500 H-B	EPA 9045C	--
	Solid		---	EPA 9045C	--
Paint Filter	Water		--	EPA 9095A	--
Phenolics	Water		EPA 420.1	--	--
	Waste		--	EPA 9065	--
	Solid		--	EPA 9065	--
Phosphorus (Total)	Water		EPA 365.1	--	SM 4500 P-E
	Waste		EPA 365.1	--	--
	Solid		EPA 365.1	--	--
Sulfate (SO_4)	Water		EPA 300.0A EPA 375.4[2]	EPA 9056A EPA 9038	--
	Waste		EPA 300.0A (M) EPA 375.4[2]	EPA 9056A EPA 9038	--
	Solid		EPA 300.0A (M)	EPA 9056A EPA 9038 (M)	--

Analytical Parameters	Matrix		Fields of Testing		
			CWA	RCRA	Other
Sulfide	Water		EPA 376.1[2]	EPA 9030A SM 4500	9030B/9034
Total Organic Carbon (TOC)	Water		EPA 415.1[2]	EPA 9060	SM 5310 D
	Waste		--	EPA 9060	--
	Solid		EPA 415.1 (M)	EPA 9060 (M)	Walkley-Black
Total Organic Halides (TOX)	Water		--	EPA 9020B EPA 9023(EOX)	EPA 450.1
	Waste		--	--	--
	Solid		--	EPA 9020B	--
Total Petroleum Hydrocarbons	Water		EPA 1664A (SGT-HEM)	EPA 9071B	--
	Waste		EPA 1664A (SGT-HEM)	EPA 9071B	--
	Solid		--	EPA 9071B	--
Total Solids	Water		EPA 160.3	--	--
	Waste		EPA 160.3	--	--
	Solid		EPA 160.3 (M)	--	--
Total Dissolved Solids	Water		EPA 160.1	--	2540E
Total Suspended Solids	Water		EPA 160.2	---	2540E
Volatile and Volatile Suspended Solids	Water		EPA 160.4	--	--
Settleable Solids	Water		EPA 160.5	--	--
Turbidity	Water		EPA 180.1	--	--

Methods for Mercury by Cold Vapor Atomic Absorption

Analytical Parameters	Matrix		Fields of Testing		
			CWA	RCRA (SW846)	Other
Mercury (CVAA)	Water		EPA 245.1	EPA 7470A	--
	TCLP Leachate		--	EPA 7470A	--
	Waste		--	EPA 7471A	--
	Solid		EPA 254.5	EPA 7471A	--

Methods for Mercury by Cold Vapor Atomic Fluororescence

Analytical Parameters	Matrix		Fields of Testing		
			CWA	RCRA (SW846)	Other
Mercury, Low Level (CVAFS)	Water		--	--	EPA 1631E

Methods for Metals by ICP and ICPMS

Analytical Parameters	Matrix		Fields of Testing		
			CWA	RCRA (SW846)	Other
Metals by ICP analysis	Water		EPA 200.7	EPA 6010B	---
	Waste		---	EPA 6010B	---
	Solid		EPA 200.7	EPA 6010B	---
Metals by ICPMS analysis	Water		EPA 200.8	EPA 6020	---
	Waste		---	EPA 6020	---
	Solid		EPA 200.8	EPA 6020	---

Metals Sample Preparation Methods

Analytical Parameters	Matrix		Fields of Testing		
			CWA	RCRA (SW846)	Other
Toxicity Characteristic Leaching Procedure (TCLP)	Water		---	EPA 1311	---
	Waste		---	EPA 1311	---
	Solid		---	EPA 1311	---
ICP Metals	Water		EPA 200.7	EPA 3005A EPA 3010A	---
	TCLP Leachate		---	EPA 3010A	---
	Waste		---	EPA 3050B	---
	Solid		---	EPA 3050B	---
ICPMS Metals	Water		EPA 200.8	EPA 3010A	---
	TCLP		---	EPA 3010A	---
	Waste		---	EPA 3050B	---
	Solid		---	EPA 3050B	---
CVAA Mercury	Water		EPA 245.1	EPA 7470A	---
	TCLP Leachate		---	EPA 7470A	---
	Waste		---	EPA 7471A	---
	Solid		---	EPA 7471A	
CVAFS Mercury Low Level	Water		---	---	EPA 1631E

Organic Sample Preparation Methods

Analytical Parameters	Matrix	Fields of Testing		
		CWA	RCRA (SW846)	Other
Volatiles by GC/MS	Water	EPA 624	EPA 5030B	---
	Waste	---	EPA 5030B EPA 5035	---
	Solid	---	EPA 5035 EPA 5035A	---
Halogenated Volatiles by GC	Water	EPA 601	EPA 5030B	---
	Waste	---	EPA 5030B EPA 5035	---
	Solid	---	EPA 5035 EPA 5035A	---
Aromatic Volatiles by GC	Water	EPA 602	EPA 5030B	---
	Waste	---	EPA 5030B EPA 5035	---
	Solid	---	EPA 5035 EPA 5035A	---
Semivolatiles by GC/MS	Water	EPA 625	EPA 3510C EPA 3520C	---
	TCLP Leachate	---	EPA 3510C EPA 3520C	---
	Waste	---	EPA 3550B EPA 3540C EPA 3580A EPA 3541	---
	Solid	---	EPA 3550B EPA 3540C EPA 3541	---
Pesticides/PCBs by GC	Water	EPA 608	EPA 3510C EPA 3520C	---
	TCLP Leachate	---	EPA 3510C EPA 3520C	---
	Waste	---	EPA 3550B EPA 3540C EPA 3580A EPA 3541	---
	Solid	---	EPA 3550B EPA 3540C EPA 3541	---

Analytical Parameters	Matrix	Fields of Testing		
		CWA	RCRA (SW846)	Other
Herbicides by GC	Water	EPA 615	EPA 8151A	---
	Waste	---	EPA 8151A	---
	Solid	---	EPA 8151A	---
Total Petroleum Hydrocarbons (Gasoline Range) by GC	Water	---	EPA 5030B	WI GRO
	Waste	---	EPA 5030B EPA 5035	WI GRO
	Solid	---	EPA 5035 EPA 5035	WI GRO
Total Petroleum Hydrocarbons (Diesel Range) by GC	Water	---	EPA 3510C EPA 3520C	WI DRO
	TCLP Leachate	---	EPA 3510C EPA 3520C	---
	Waste	---	EPA 3550B EPA 3580A	WI DRO
	Solid	---	EPA 3550B	WI DRO

Organic Methods of Analysis

Analytical Parameters	Matrix		Fields of Testing		
			CWA	RCRA (SW846)	Other
Volatiles by GC/MS	Water		EPA 624	EPA 8260B	---
	Waste		---	EPA 8260B	---
	Solid		---	EPA 8260B	---
Halogenated Volatiles by GC	Water		EPA 601	EPA 8021B	---
	Waste		---	EPA 8021B	---
	Solid		---	EPA 8021B	---
Aromatic Volatiles by GC	Water		EPA 602	EPA 8021B	---
	Waste		---	EPA 8021B	---
	Solid		---	EPA 8021B	---
Semivolatiles by GC/MS	Water		EPA 625	EPA 8270C	
	Waste		---	EPA 8270C	---
	Solid		---	EPA 8270C	---
Pesticides/PCBs by GC	Water		EPA 608	Pesticides 8081A PCBs 8082	---
	TCLP Leachate		---	Pesticides 8081A PCBs 8082	---
	Waste		---	Pesticides 8081A PCBs 8082	---
	Solid		---	Pesticides 8081A PCBs 8082	---

Analytical Parameters	Matrix		Fields of Testing		
			CWA	RCRA (SW846)	Other
Phenoxyacid Herbicides by GC	Water		---	EPA 8151A	---
	TCLP Leachate		---	EPA 8151A	---
	Waste		---	EPA 8151A	---
	Solid		---	EPA 8151A	---
Gasoline Range Organics by GC	Water		---	EPA 8015B (M)	WI GRO
	Waste		---	EPA 8015B (M)	---
	Solid		---	EPA 8015B (M)	WI GRO
Total Petroleum Hydrocarbons (Diesel Range) by GC/FID	Water		---	EPA 8015B (M)	WI DRO
	Waste		---	EPA 8015B (M)	---
Dissolved Gases RSK-175	Water		---	---	SOP
Formaldehyde Carbonyl Compounds	Water		---	EPA 8315	---

Appendix 3. Glossary/Acronyms

Glossary

Acceptance Criteria:
Specified limits placed on characteristics of an item, process, or service defined in requirement documents. (ASQC)

Accreditation:
The process by which an agency or organization evaluates and recognizes a laboratory as meeting certain predetermined qualifications or standards, thereby accrediting the laboratory. In the context of the National Environmental Laboratory Accreditation Program (NELAP), this process is a voluntary one. (NELAC)

Accrediting Authority:
The Territorial, State, or Federal Agency having responsibility and accountability for environmental laboratory accreditation and which grants accreditation (NELAC) [1.5.2.3]

Accuracy:
The degree of agreement between an observed value and an accepted reference value. Accuracy includes a combination of random error (precision) and systematic error (bias) components which are due to sampling and analytical operations; a data quality indicator. (QAMS)

Analyst:
The designated individual who performs the "hands-on" analytical methods and associated techniques and who is the one responsible for applying required laboratory practices and other pertinent quality controls to meet the required level of quality. (NELAC)

Assessment:
The evaluation process used to measure or establish the performance, effectiveness, and conformance of an organization and/or its systems to defined criteria (to the standards and requirements of NELAC). (NELAC)

Assessment Criteria:
The measures established by NELAC and applied in establishing the extent to which an applicant is in conformance with NELAC requirements. (NELAC)

Assessment Team:
The group of people authorized to perform the on-site inspection and proficiency testing data evaluation required to establish whether an applicant meets the criteria for NELAP accreditation. (NELAC)

Assessor:
One who performs on-site assessments of accrediting authorities and laboratories' capability and capacity for meeting NELAC requirements by examining the records and other physical evidence for each one of the tests for which accreditation has been requested. (NELAC)

Audit:
A systematic evaluation to determine the conformance to quantitative and qualitative specifications of some operational function or activity.

Batch:
Environmental samples which are prepared and/or analyzed together with the same process and personnel, using the same lot(s) of reagents. A preparation batch is composed of one to 20 environmental samples of the same matrix, meeting the above mentioned criteria and with a maximum time between the start of processing of the first and last sample in the batch to be 24 hours. An analytical batch is composed of prepared environmental samples (extracts, digestates or concentrates) and /or those samples not requiring preparation, which are analyzed together as a group using the same calibration curve or factor. An analytical batch can include samples originating from various environmental matrices and can exceed 20 samples. (NELAC Quality Systems Committee)

Blank:
A sample that has not been exposed to the analyzed sample stream in order to monitor contamination during sampling, transport, storage or analysis. The blank is subjected to the usual analytical and measurement process to establish a zero baseline or background value and is sometimes used to adjust or correct routine analytical results. (ASQC)

Blind Sample:
A sample for analysis with a composition known to the submitter. The analyst/laboratory may know the identity of the sample but not its composition. It is used to test the analyst's or laboratory's proficiency in the execution of the measurement process.

Calibration:
To determine, by measurement or comparison with a standard, the correct value of each scale reading on a meter, instrument, or other device. The levels of the applied calibration standard should bracket the range of planned or expected sample measurements. (NELAC)

Calibration Curve:
The graphical relationship between the known values, such as concentrations, of a series of calibration standards and their instrument response. (NELAC)

Calibration Method:
A defined technical procedure for performing a calibration. (NELAC)

Calibration Standard:
A substance or reference material used to calibrate an instrument (QAMS)

Certified Reference Material (CRM):
A reference material one or more of whose property values are certified by a technically valid procedure, accompanied by or traceable to a certificate or other documentation which is issued by a certifying body. (ISO Guide 30–2.2)

Chain-of-Custody:
An unbroken trail of accountability that ensures the physical security of samples and includes the signatures of all who handle the samples. (NELAC) [5.12.4]

Clean Air Act:
The enabling legislation in 42 U>S>C> 7401 et seq., Public Law 91-604, 84 Stat. 1676 Pub. L. 95-95, 91 Stat., 685 and Pub. L. 95-190, 91 Stat., 1399, as amended, empowering EPA to promulgate air quality standards, monitor and enforce them. (NELAC)

Comprehensive Environmental Response, Compensation and Liability Act (CERCLA/SUPERFUND):
The enabling legislation in 42 U.S.C. 9601-9675 et seq., as amended by the Superfund Amendments and Reauthorization Act of 1986 (SARA), 42 U.S.C. 9601 et seq., to eliminate the health and environmental threats posed by hazardous waste sites. (NELAC)

Compromised Samples:
Those samples which are improperly sampled, insufficiently documented (chain of custody and other sample records and/or labels), improperly preserved, collected in improper containers, or exceeding holding times when delivered to a laboratory. Under normal conditions, compromised samples are not analyzed. If emergency situation require analysis, the results must be appropriately qualified. (NELAC)

Confidential Business Information (CBI):
Information that an organization designates as having the potential of providing a competitor with inappropriate insight into its management, operation or products. NELAC and its representatives agree to safeguarding identified CBI and to maintain all information identified as such in full confidentiality.

Confirmation:
Verification of the identity of a component through the use of an approach with a different scientific principle from the original method. These may include, but are not limited to:

Second column confirmation
Alternate wavelength
Derivatization
Mass spectral interpretation
Alternative detectors or
Additional Cleanup procedures
(NELAC)

Conformance:
An affirmative indication or judgment that a product or service has met the requirements of the relevant specifications, contract, or regulation; also the state of meeting the requirements. (ANSI/ASQC E4-1994)

Correction*:*
Actions necessary to correct or repair analysis specific non-conformances. The acceptance criteria for method specific QC and protocols as well as the associated corrective actions. The analyst will most frequently be the one to identify the need for this action as a result of calibration checks and QC sample analysis**.** No significant action is taken to change behavior, process or procedure.

Corrective Action:
The action taken to eliminate the causes of an existing nonconformity, defect or other undesirable situation in order to prevent recurrence. (ISO 8402)

Data Audit:
A qualitative and quantitative evaluation of the documentation and procedures associated with environmental measurements to verify that the resulting data re of acceptable quality (i.e., that they meet specified acceptance criteria). (NELAC)

Data Reduction:
The process of transforming raw data by arithmetic or statistical calculations, standard curves, concentration factors, etc., and collation into a more useable form. (EPA-QAD)

Deficiency:
An unauthorized deviation from acceptable procedures or practices, or a defect in an item. (ASQC)

Detection Limit:
The lowest concentration or amount of the target analyte that can be identified, measured, and reported with confidence that the analyte concentration is not a false positive value. See Method Detection Limit. (NELAC)

Document Control:
The act of ensuring that documents (and revisions thereto) are proposed, reviewed for accuracy, approved for release by authorized personnel, distributed properly, and controlled to ensure use of the correct version at the location where the prescribed activity if performed. (ASQC)

Duplicate Analyses:
The analyses or measurements of the variable of interest performed identically on two subsamples of the same sample. The results from duplicate analyses are used to evaluate analytical or measurement precision but not the precision of sampling, preservation or storage internal to the laboratory. (EPA-QAD)

Equipment Blank:
Sample of analyte-free media which has been used to rinse common sampling equipment to check effectiveness of decontamination procedures. (NELAC)

External Standard Calibration:
Calibrations for methods that do not utilize internal standards to compensate for changes in instrument conditions.

Federal Insecticide, Fungicide and Rodenticide Act (FIFRA):
The enabling legislation under 7 U.S.C. 135 et seq., as amended, that empowers the EPA to register insecticides, fungicides, and rodenticides. (NELAC)

Federal Water Pollution Control Act (Clean Water Act, CWA):
The enabling legislation under 33 U.S.C. 1251 et seq., Public Law 92-50086 Stat 816, that empowers EPA to set discharge limitations, write discharge permits, monitor, and bring enforcement action for non-compliance. (NELAC)

Field Blank:
Blank prepared in the field by filing a clean container with pure de-ionized water and appropriate preservative, if any, for the specific sampling activity being undertaken (EPA OSWER)

Field of Testing:
NELAC's approach to accrediting laboratories by program, method and analyte. Laboratories requesting accreditation for a program-method-analyte combination or for an up-dated/improved method are required to submit to only that portion of the accreditation process not previously addressed (see NELAC, section 1.9ff). (NELAC)

Finding:
An assessment conclusion that identifies a condition having a significant effect on an item or activity. As assessment finding is normally a deficiency and is normally accompanied by specific examples of the observed condition. (NELAC)

Holding Times (Maximum Allowable Holding Times):
The maximum times that samples may be held prior to analyses and still be considered valid or not compromised. (40 CFR Part 136)

Inspection:
An activity such as measuring, examining, testing, or gauging one or more characteristics of an entity and comparing the results with specified requirements in order to establish whether conformance is achieved for each characteristic. (ANSI/ASQC E4-1994)

Internal Standard:
A known amount of standard added to a test portion of a sample and carried through the entire measurement process as a reference for evaluating and controlling the precision and bias of the applied analytical test method. (NELAC)

Internal Standard Calibration:
Calibrations for methods that utilize internal standards to compensate for changes in instrument conditions.

Instrument Blank:
A clean sample (e.g., distilled water) processed through the instrumental steps of the measurement process; used to determine instrument contamination. (EPA-QAD)

Instrument Response:
Instrument response is normally expressed as either peak area or peak height however it may also reflect a numerical representation of some type of count on a detector (e.g. Photomultiplier tube, or Diode array detector) and is used in this document to represent all types.

Laboratory:
A defined facility performing environmental analyses in a controlled and scientific manner. (NELAC)

Laboratory Control Sample (however named, such as laboratory fortified blank, spiked blank, or QC check sample):
A sample matrix, free from the analytes of interest, spiked with verified known amounts of analytes or a material containing known and verified amounts of analytes, taken through all preparation and analysis steps. Where there is no preparation taken for an analysis (such as in aqueous volatiles), or when all samples and standards undergo the same preparation and analysis process (such as Phosphorus), there is no LCS. It is generally used to establish intra-laboratory or analyst specific precision and bias or to assess the performance of all or a portion of the measurement system.

An LCS must be prepared at a minimum of 1 per batch of 20 or less samples per matrix type per sample extraction or preparation method except for analytes for which spiking solutions are not available such as total suspended solids, total dissolved solids, total volatile solids, total solids, pH, color, odor, temperature, dissolved oxygen or turbidity. The results of these samples must be used to determine batch acceptance.

Note: NELAC standards allow a matrix spike to be used in place of this control as long as the acceptance criteria are as stringent as for the LCS. (NELAC)

Laboratory Duplicate:
Aliquots of a sample taken from the same container under laboratory conditions and processed and analyzed independently. (NELAC)

Least Squares Regression (1st Order Curve):
The least squares regression is a mathematical calculation of a straight line over two axes. The y axis represents the instrument response (or Response ratio) of a standard or sample and the x axis represents the concentration. The regression calculation will generate a correlation coefficient (r) that is a measure of the "goodness of fit" of the regression line to the data. A value of 1.00 indicates a perfect fit. In order to be used for quantitative purposes, r must be greater than or equal to 0.99 for organics and 0.995 for inorganics.

Limit of Detection (LOD):
An estimate of the minimum amount of a substance that an analytical process can reliably detect. An LOD is analyte- and matrix-specific and may be laboratory dependent. (Analytical Chemistry, 55, p.2217, December 1983, modified) See also Method Detection Limit.

Matrix:
The component or substrate that contains the analyte of interest. For purposes of batch and QC requirement determinations, the following matrix distinctions must be used:

> Aqueous: Any aqueous sample excluded from the definition of Drinking Water matrix or Saline/Estuarine source. Includes surface water, groundwater, effluents, and TCLP or other extracts.
>
> Drinking Water: any aqueous sample that has been designated as a potable or potential potable water source.
>
> Saline/Estuarine: any aqueous sample from an ocean or estuary, or other salt water source such as the Great Salt Lake.

Non-aqueous Liquid: any organic liquid with ,15% settleable solids.

Biological Tissue: any sample of a biological origin such as fish tissue, shellfish, or plant material. Such samples must be grouped according to origin.

Solids: includes soils, sediments, sludges, and other matrices with .15% settleable solids.

Chemical Waste: a product or by-product of an industrial process that results in a matrix not previously defined.

Air: whole gas or vapor samples including those contained in flexible or rigid wall containers and the extracted concentrated analytes of interest from a gas or vapor that are collected with a sorbant tube, impinger solution, filter, or other device. (NELAC)

Matrix Spike (spiked sample or fortified sample):
Prepared by adding a known mass of target analyte to a specified amount of matrix sample for which an independent estimate of target analyte concentration is available. Matrix spikes are used, for example, to determine the effect of the matrix on a method's recovery efficiency. Matrix spikes must be performed at a frequency of one in 20 samples per matrix type per sample extraction or preparation method except for analytes for which spiking solutions are not available such as, total suspended solids, total dissolved solids, total volatile solids, total solids, pH, color, odor, temperature, dissolved oxygen or turbidity. The selected sample(s) must be rotated among client samples so that various matrix problems may be noted and/or addressed. Poor performance in a matrix spike may indicate a problem with the sample composition and must be reported to the client whose sample was used for the spike. (QAMS)

Matrix Spike Duplicate (spiked sample or fortified sample duplicate):
A second replicate matrix spike is prepared in the laboratory and analyzed to obtain a measure of the precision of the recovery for each analyte. Matrix spike duplicates or laboratory duplicates must be analyzed at a minimum of 1 in 20 samples per matrix type per sample extraction or preparation method. The laboratory must document their procedure to select the use of an appropriate type of duplicate. The selected sample(s) must be rotated among client samples so that various matrix problems may be noted and/or addressed. Poor performance in the duplicates may indicate a problem with the sample composition and must be reported to the client whose sample was used for the duplicate. (QAMS)

Method Blank:
A sample of a matrix similar to the batch of associated samples (when available) that is free from the analytes of interest and is processed simultaneously with and under the same conditions as samples through all steps of the analytical procedures, and in which no target analytes or interferences are present at concentrations that impact the analytical results for sample analyses. (NELAC)

Method Detection Limit:
The minimum concentration of a substance (an analyte) that can be measured and reported with 99% confidence that the analyte concentration is greater than zero and is determined from analysis of a sample in a given matrix containing the analyte. (40 CFR Part 136, Appendix B)

National Environmental Laboratory Accreditation Conference (NELAC):
A voluntary organization of State and Federal environmental officials and interest groups purposed primarily to establish mutually acceptable standards for accrediting environmental laboratories. A subset of NELAP. (NELAC)

National Environmental Laboratory Accreditation Program (NELAP):
The overall National Environmental Laboratory Accreditation Program of which NELAC is a part. (NELAC)

Negative Control:
Measures taken to ensure that a test, its components, or the environment do not cause undesired effects, or produce incorrect test results. (NELAC)

NELAC Standards:
The plan of procedures for consistently evaluating and documenting the ability of laboratories performing environmental measurements to meet nationally defined standards established by the National Environmental Laboratory Accreditation Conference. (NELAC)

Performance Audit:
The routine comparison of independently obtained qualitative and quantitative measurement system data with routinely obtained data in order to evaluate the proficiency of an analyst or laboratory. (NELAC)

Performance Based Measurement System (PBMS):
A set of processes wherein the data quality needs, mandates or limitations of a program or project are specified and serve as criteria for selecting appropriate test methods to meet those needs in a cost-effective manner. (NELAC)

Positive Control:
Measures taken to ensure that a test and/or its components are working properly and producing correct or expected results from positive test subjects. (NELAC)

Precision:
The degree to which a set of observations or measurements of the same property, obtained under similar conditions, conform to themselves; a data quality indicator. Precision is usually expressed as standard deviation, variance or range, in either absolute or relative terms. (NELAC)

Preservation:
Refrigeration and/or reagents added at the time of sample collection (or later) to maintain the chemical and/or biological integrity of the sample. (NELAC)

Proficiency Testing:
A means of evaluating a laboratory's performance under controlled conditions relative to a given set of criteria through analysis of unknown samples provided by an external source. (NELAC) [2.1]

Proficiency Testing Program:
The aggregate of providing rigorously controlled and standardized environmental samples to a laboratory for analysis, reporting of results, statistical evaluation of the results and the collective demographics and results summary of all participating laboratories. (NELAC)

Proficiency Test Sample (PT):
A sample, the composition of which is unknown to the analyst and is provided to test whether the analyst/laboratory can produce analytical results within specified acceptance criteria. (QAMS)

Quality Assurance:
An integrated system of activities involving planning, quality control, quality assessment, reporting and quality improvement to ensure that a product or service meets defined standards of quality with a stated level of confidence. (QAMS)

Quality Assurance [Project] Plan (QAPP):
A formal document describing the detailed quality control procedures by which the quality requirements defined for the data and decisions pertaining to a specific project are to be achieved. (EAP-QAD)

Quality Control:
The overall system of technical activities which purpose is to measure and control the quality of a product or service so that it meets the needs of users. (QAMS)

Quality Control Sample:
An uncontaminated sample matrix spiked with known amounts of analytes from a source independent from the calibration standards. It is generally used to establish intra-laboratory or analyst specific precision and bias or to assess the performance of all or a portion of the measurement system. (EPA-QAD)

Quality Manual:
A document stating the management policies, objectives, principles, organizational structure and authority, responsibilities, accountability, and implementation of an agency, organization, or laboratory, to ensure the quality of its product and the utility of its product to its users. (NELAC)

Quality System:
A structured and documented management system describing the policies, objectives, principles, organizational authority, responsibilities, accountability, and implementation plan of an organization for ensuring quality in its work processes, products (items), and services. The quality system provides the framework for planning, implementing, and assessing work performed by the organization and for carrying out required QA and QC (ANSI/ASQC-E-41994)

Quantitation Limits:
The maximum or minimum levels, concentrations, or quantities of a target variable (e.g., target analyte) that can be quantified with the confidence level required by the data user. (NELAC)

Range:
The difference between the minimum and the maximum of a set of values. (EPA-QAD)

Reagent Blank (method reagent blank):
A sample consisting of reagent(s), without the target analyte or sample matrix, introduced into the analytical procedure at the appropriate point and carried through all subsequent steps to determine the contribution of the reagents and of the involved analytical steps. (QAMS)

Reference Material:
A material or substance one or more properties of which are sufficiently well established to be used for the calibration of an apparatus, the assessment of a measurement method, or for assigning values to materials. (ISO Guide 30-2.1)

Reference Method:
A method of known and documented accuracy and precision issued by an organization recognized as competent to do so. (NELAC)

Reference Standard:
A standard, generally of the highest metrological quality available at a given location, from which measurements made at that location are derived. (VIM-6.0-8)

Replicate Analyses:
The measurements of the variable of interest performed identically on two or more sub-samples of the same sample within a short time interval. (NELAC)

Report Limit (RL):
The laboratory nominal Quantitation Limit (QL) or the level of sensitivity required by the client but not lower than the LOD. Reporting limits are easily accessible via LIMS (QC Browser program).

Requirement:
Denotes a mandatory specification; often designated by the term “shall”. (NELAC)

Resource Conservation and Recovery Act (RCRA):
The enabling legislation under 42 USC 321 et seq. (1976), that gives EPA the authority to control hazardous waste from the “cradle-to-grave”, including its generation, transportation, treatment, storage, and disposal. (NELAC)

Safe Drinking Water Act (SDWA):
The enabling legislation, 42 USC 300f et seq. (1974), (Public Law 93-523), that requires the EPA to protect the quality of drinking water in the U.S. by setting maximum allowable contaminant levels, monitoring, and enforcing violations. (NELAC)

Sample Duplicate:
Two samples taken from and representative of the same population and carried through all steps of the sampling and analytical procedures in an identical manner. Duplicate samples are used to assess variance of the total method including sampling and analysis. (EPA-QAD)

Second Order Polynomial Curve (Quadratic): The 2^{nd} order curves are a mathematical calculation of a slightly curved line over two axis. The y axis represents the instrument response (or Response ratio) of a standard or sample and the x axis represents the concentration. The 2^{nd} order regression will generate a coefficient of determination (COD or r^2) that is a measure of the "goodness of fit" of the quadratic curvature the data. A value of 1.00

indicates a perfect fit. In order to be used for quantitative purposes, r^2 must be greater than or equal to 0.99.

Selectivity:
(Analytical chemistry) the capability of a test method or instrument to respond to a target substance of constituent in the presence of non-target substances. (EPA-QAD)

Sensitivity:
The capability of a method or instrument to discriminate between measurement responses representing different levels (e.g., concentrations) of a variable of interest. (NELAC)

Spike:
A known mass of target analyte added to a blank, sample or sub-sample; used to determine recovery efficiency or for other quality control purposes.

If the mandated or requested test method does not specify the spiking components, the laboratory shall spike all reportable components to be reported in the Laboratory Control Sample and Matrix Spike. However, in cases where the components interfere with accurate assessment (such as simultaneously spiking chlordane, toxaphene and PCBs in Method 608), the test method has an extremely long list of components or components are incompatible, a representative number (at a minimum 10%) of the listed components may be used to control the test method. The selected components of each spiking mix shall represent all chemistries, elution patterns and masses permit specified analytes and other client requested components. However, the laboratory shall ensure that all reported components are used in the spike mixture within a two-year time period. (NELAC)

Standard:
The document describing the elements of laboratory accreditation that has been developed and established within the consensus principles of NELAC and meets the approval requirements of NELAC procedures and policies. (ASQC)

Standard Operating Procedures (SOPs):
A written document which details the method of an operation, analysis, or action whose techniques and procedures are thoroughly prescribed and which is accepted as the method for performing certain routine or repetitive tasks. (QAMS)

Standardized Reference Material (SRM):
A certified reference material produced by the U.S. National Institute of Standards and Technology or other equivalent organization and characterized for absolute content, independent of analytical method. (EPA-QAD)

Supervisor (however named):
The individual(s) designated as being responsible for a particular area or category of scientific analysis. This responsibility includes direct day-to-day supervision of technical employees, supply and instrument adequacy and upkeep, quality assurance/quality control duties, and ascertaining that technical employees have the required balance of education, training and experience to perform the required analyses. (NELAC)

Surrogate:
A substance with properties that mimic the analyte of interest. It is unlikely to be found in environment samples and is added to them for quality control purposes.

Surrogate compounds must be added to all samples, standards, and blanks, for all organic chromatography methods except when the matrix precludes its use or when a surrogate is not available. Poor surrogate recovery may indicate a problem with sample composition and must be reported to the client whose sample produced poor recovery. (QAMS)

Systems Audit (also Technical Systems Audit):
A thorough, systematic, qualitative on-site assessment of the facilities, equipment, personnel, training, procedures, record keeping, data validation, data management, and reporting aspects of a total measurement system. (EPA-QAD)

Technical Director:
Individuals(s) who has overall responsibility for the technical operation of the environmental testing laboratory. (NELAC)

Test:
A technical operation that consists of the determination of one or more characteristics or performance of a given product, material, equipment, organism, physical phenomenon, process, or service according to a specified procedure. The result of a test is normally recorded in a document sometimes called a test report or a test certificate. (ISO/IEC Guide 2-12.1, amended)

Test Method:
An adoption of a scientific technique for a specific measurement problem, as documented in a laboratory SOP. (NELAC)

Toxic Substances Control Act (TSCA):
The enabling legislation in 15 USC 2601 et seq., (1976) that provides for testing, regulating, and screening all chemicals produced or imported into the United States for possible toxic effects prior to commercial manufacture. (NELAC)

Traceability:
The property of a result of a measurement whereby it can be related to appropriate standards, generally international or national standards, through an unbroken chain of comparisons. (VIM-6.12)

Uncertainty:
A parameter associated with the result of a measurement that characterizes the dispersion of the value that could reasonably be attributed to the measured value.

United States Environmental Protection Agency (EPA):
The Federal governmental agency with responsibility for protecting public health and safeguarding and improving the natural environment (i.e., the air, water, and land) upon which human life depends. (US-EPA)

Validation:
The process of substantiating specified performance criteria. (EPA-QAD)

Verification:
Confirmation by examination and provision of evidence that specified requirements have been met. (NELAC)

Note: In connection with the management of measuring equipment, verification provides a means for checking that the deviations between values indicated by a measuring instrument and corresponding known values of a measured quantity are consistently smaller than the maximum allowable error defined in a standard, regulation or specification peculiar to the management of the measuring equipment. The result of verification leads to a decision either to restore in service, to perform adjustment, to repair, to downgrade, or to declare obsolete. In all cases, it is required that a written trace of the verification performed must be kept on the measuring instrument's individual record.

Acronyms

BS	Blank Spike
BSD	Blank Spike Duplicate
CAR	Corrective Action Report
CCV	Continuing Calibration Verification
CF	Calibration Factor
CFR	Code of Federal Regulations
COC	Chain of Custody
CRS	Change Request Form
DOC	Demonstration of Capability
DQO	Data Quality Objectives
DU	Duplicate
DUP	Duplicate
EHS	Environment, Health and Safety
EPA	Environmental Protection Agency
GC	Gas Chromatography
GC/MS	GasChromatography/Mass Spectrometry
HPLC	High Performance Liquid Chromatography
ICP	Inductively Coupled Plasma Atomic Emission Spectroscopy
ICV	Initial Calibration Verification
IDL	Instrument Detection Limit
IH	Industrial Hygiene
IS	Internal Standard
LCS	Laboratory Control Sample
LCSD	Laboratory Control Sample Duplicate
LIMS	Laboratory Information Management System
MDL	Method Detection Limit
MS	Matrix Spike
MSD	Matrix Spike Duplicate
MSDS	Material Safety Data Sheet
NELAC	National Environmental Laboratory Accreditation Conference
NELAP	National Environmental Laboratory Accreditation Program
PT	Performance Testing
QAM	Quality Assurance Manual
QA/QC	Quality Assurance / Quality Control
QAPP	Quality Assurance Project Plan
RF	Response Factor
RPD	Relative Percent Difference
RSD	Relative Standard Deviation
SD	Standard Deviation
SOP	Standard Operating Procedure
TAT	Turn-Around Time
VOA	Volatiles
VOC	Volatile organic Compound

Company Confidential & Proprietary

Appendix 4. Laboratory Certifications, Accreditations, Validations

TestAmerica North Canton maintains certifications, accreditations, certifications, and validations with numerous state and national entities. Programs vary but may include on-site audits, reciprocal agreements with another entity, performance testing evaluations, review of the QA Manual, Standard Operating Procedures, Method Detection Limits, training records, etc. At the time of this QA Manual revision, the laboratory has accreditation/certification/licensing with the following organizations:

Organization	Certificate Number	Organization	Certificate Number
California	01144CA	Nevada	OH-00048208A
Connecticut	PH-0590	New Jersey	OH001
Florida	E87225	New York	10975
Georgia	---	OVAP	CL0024
Illinois	001298	Pennsylvania	68-00340
Kansas	E-10336	US ACE (Army)	---
Kentucky Underground Storage Tank Program	0058	USDA (Dept.of Agriculture)	P330-08-00123
Minnesota	039-999-348	West Virginia	210
DoD - ACLASS	ADE-1437---	Wisconsin	999518190

The certificates and parameter lists (which may differ) for each organization may be found on the corporate web site, the laboratory's public server, the final report review table, and in the following offices: QA, Marketing, and Project Management.

APPENDIX B

Field Analytical Standard Operating Procedures

pH/TEMPERATURE

Scope and Application: This method is applicable to surface water, wastewater and groundwater.

Method: Potentiometric.

Reference: "Methods for Chemical Analysis of Water and Wasters", EPA-600/4-79-020, revised March 1983, Method 150.1.

Sensitivity: 0.01 pH unit; 0.1^{o}C.

Optimum Range: pH 1.00 to 12.00; temperature 0 to 100 ^{o}C.

Sample Handling: determined on site.

Reagents and Apparatus:

1. Temperature compensated pH meter, Fisher Scientific Accumet Series 1000 or equivalent;
2. Combination pH electrode;
3. Temperature sensor verified against NIST traceable thermometer on an annual basis;
4. pH buffer solutions, pH 4.00, 7.00 and 10.00 (certified buffer solutions);
5. Deionized water in wash bottle.

Calibration:

1. Press On/Off key to turn meter on. If pH indicator in main display area is not on, press pH key to place meter in pH mode.

   Main display will show pH reading. If ATC probe is attached, temperature reading will appear in center display. If meter was previously standardized, buffers used will be shown in lower display.

2. Set pH display resolution by pressing Exp key until desired resolution is shown.
3. Clear current standardization points by pressing Stdz key. Then, within four seconds, press Clear key. Current standardization points will be removed from instrument memory.
4. Immerse electrodes in pH 4.00 buffer. Press Stdz key to begin standardization.
5. Rinse electrodes and immerse in pH 7.00 buffer. Press Stdz key to begin standardization with this buffer.

In lower display, nominal (25 °C) value for second buffer will begin flashing, while value for first buffer remains on. Main display will track electrode output in millivolts. When electrode output stabilizes, second buffer value will cease flashing and also remain on.

6. Rinse electrode and immerse in pH 10.00 buffer. Press Stdz key to begin standardization with this buffer.

   In lower display, nominal (25 °C) value for third buffer will begin flashing, while values for first and second buffers remain on. Main display will track electrode output in millivolts. When electrode output stabilizes, third buffer value will cease flashing and also remain on.

7. Rinse electrode with deionized water and place in pH 7.00 buffer. If meter reading is not 7.00, follow steps 4 to 7 again.

Procedure:

1. Calibrate meter using calibration procedure.

2. Pour the sample into clean sample jar or plastic cup.

3. If stability bar is not currently on, press Auto key. Otherwise, press pH key. This initiates measurement of first sample pH.

   Stability bar will flash and main display will begin to track sample pH. When both electrode output and ATC stabilize, stability bar will remain on with pH reading locked.

4. Record temperature and pH of the sample in the logbook.

5. Record steps 3 and 4 for each sample.

6. Recheck calibration with pH 7.00 buffer solution after a minimum of every 10 samples and after last sample.

Quality Control:

1. Duplicate 1 out of 10 samples. If less that 10 samples are analyzed, a duplicate is still required. Duplicates must be $\pm$0.2 pH units.

   If the results are outside of the control limits, rinse electrodes and repeat analysis. If results are still outside of the control limits, recollect samples and repeat analysis. If the results are still outside of the control limits, check calibration and recalibrate if necessary (see item 2, below). If drift is suspected to be the cause of the problem, clean the electrode and recalibrate. If drift is still apparent, replace electrode.

2. Calibration check results must be $\pm$0.10 pH unit of the true value. If the result is outside of $\pm$0.10 pH unit, rinse electrodes and check solution again. If still outside the control limit, recalibrate the meter and reanalyze all samples analyzed since the last in control calibration.

3. All glassware is to be soap and water washed, tap rinsed and deionized water rinsed prior to analyses, unless pre-cleaned sample jars are used.

Interferences:

Interferences in pH measurements occur with presence of weak organic and inorganic salts and oil and grease. If oil and grease are visible, note in logbook. Clean electrode with soap and water, followed by 10% HCI and deionized water. Then recalibrate meter before analysis of next sample.

Specific Conductance

Scope and Application: This method is applicable to surface water, wastewater and groundwater.

Method: Specific Conductance.

Reference: "Methods for Chemical Analysis of Water and Wastes" EPA-600/4-79-020, revised March 1983, Method 120.1.

Sensitivity: 10 μmhos/cm.

Optimum Range: 10-20,000 μmhos/cm.

Sample Handling: Determine on site.

Reagents and Apparatus:

1. Conductivity meter – Cambridge Scientific Industries Model 301353 or equivalent;
2. Deionized water;
3. Conductivity standard, 1,413 μmhos/cm @ 77^{o}F (0.01 M KCI).

Notes:

The conductivity meter is factory calibrated. The calibration is checked using a solution of known conductance and recalibrated, if necessary.

Recalibration:

To recalibrate conductance, remove black plug revealing the adjustment potentiometer screw. Add standard solution to cup, discard and refill. Repeat procedure until the digital display indicates the same value twice in a row. Adjust the potentiometer until the digital display indicates the known value of conductance. To increase the digital display reading, turn the adjustment potentiometer screw counterclockwise (clockwise to decrease).

Procedure:

1. Rinse the inside of sample cup with liquid to be measured (This is especially important if samples with a wide range of conductivity are to be measured).

2. Fill sample cup.

3. Fill sample cup at least 2/3 full. If the sample is hot boiler water, allow to cool to 160^{o}F or below.

4. slide the right hand function switch to "TEMP" and push the "READ" button. If temperature reading is not stable, empty and refill cup several times to bring cup and sample to the same temperature.

5. Read the temperature on the digital display panel and adjust both temperature compensation knobs accordingly.

6. If the approximate conductance is known, slide the left hand range selector switch to the proper range.

   Example: If you expect the sample to be around 2,000 μmhos, slide the lift hand selector switch to x 1,000.

7. Slide the right hand function switch to "COND" and push the "READ" button.

8. Multiply the digital display reading by the factor indicated by the position of the left hand range switch to determine conductance.

   Example: A display reading of 1.00 with the left hand range selector switch indicating x 1,000 is:

   1.00 x 1,000 or 1,000 μmhos/cm.

   Note: If a single "1" appears on the left hand side of the digital display, the sample conductance is higher than the selected range. Slide the left hand (range) selector switch in one step intervals until a 3 or 4 digit display appears.

   Conversely, if decimal display appears (such as 0.11) move the range selector switch to the left until a 3 or 4 digit number, 1.00 or larger, appears o the display. This puts the unit in a range affording the best accuracy. Caution: A single "1" always means that the conductance is higher than the selected range.

9. Repeat steps 1 through 8 for remaining samples.

Quality Control:

1. the quality control calibration check standard must be analyzed initially, after a minimum of every 10 samples and after the last sample. If less than 10 samples are analyzed, the calibration standards are still required. The standards must be within ±10 percent of the true value or the samples run after the last acceptable check standard are to be reanalyzed. Record the calibration standard in the field logbook.

2. Duplicate a minimum of 1 out of 10 samples. If less than 10 samples are analyzed, a duplicate is still required. Duplicate values are to be within ±15% of each other. If outside of this range, reanalyze the samples. If still outside the acceptance range, recollect sample and reanalyze. If still out, replace meter.

SOIL VOC SCREENING

Scope and Application: This method is applicable to screening VOCs in the headspace of soil samples.

Method; Photoionization

Sensitivity: Approximately 0.5 ppm depending on background

Optimum Range: background to 2,000 ppm

Reagents and Apparatus:

1. HNu Model PI-101 Photoionizer or Photovac MicroTIP Photoionization Detector;

2. Calibration gas;

3. Calibration apparatus and tubing;

4. Battery rechargers.

Calibration:

A. Calibration of the HNu

1. Connect the analyzer to the regulator and calibration has cylinder with a short segment (butt connection) of tubing. The calibration gas consists of a mixture of isobutylene and zero air. isobutylene is non toxic and safe to use in confined areas. There are no listed exposure levels at any concentration.

   It is important that the tubing be clean since contaminated tubing will affect the calibration reading. A cylinder containing less than 30 psig pressure will not be used, as readings below that level can deviate up to ten percent from the rated value.

   Turn the function switch to the STANDBY position. Turn the zero knob until the meter needle rests at 0.

2. With the SPAN setting and the function switch at the same positions as listed in the Application Data Sheet or Calibration Report, open the valve on the cylinder until a steady reading is obtained.

3. If the reading is the same as the recorded data, the analyzer calibration for the original species of interest is still correct.

4. If the reading has changed, adjust the SPAN setting until the reading is the same.

5. Shut off the cylinder as soon as the reading is established.

6. Record and maintain this new SPAN setting.

7. Background organic vapor readings should be measured prior to any soil screening.

B. Calibration of MicroTip

1. Connect the supplied regulator to the span gas cylinder. Hand tighten the fittings. Observe proper handling techniques for all gases.

2. Open the valve on the gas bag by turning the valve stem fully counterclockwise.

3. Attach the nut to the regulator. Hand tighten the fittings.

4. Turn the regulator knob counterclockwise about half a turn to start the flow of gas.

5. Fill the gas bag about half full and then close the regulator fully clockwise to turn off the flow of gas.

6. Disconnect the bag from the adapter and empty it. Flush the bag a few times with the span gas and then fill it.

7. Close the gas bag by turning the valve clockwise.

8. Press SETUP and select the desired Cal Memory with the arrow keys and press ENTER. Press EXIT to leave Setup.

9. Press CAL and enter the desired response factor. Use Table 2 from the manual to find the correct response factor. If the compound is not in Table 2 or you are not looking specifically for one compound, then enter 1.00.

   The concentration detected by MicroTIP will be multiplied by the response factor before it is displayed and logged.

10. Expose MicroTIP to zero air. Press ENTER and MicroTIP sets its zero point.

11. MicroTIP then asks for the span gas concentration. Enter the known span gas concentration and then connect the span gas bag adapter to the inlet.

12. Press ENTER and MicroTIP sets its sensitivity.

13. When MicroTIP's display reverts to normal, MicroTIP is calibrated and ready for use. Remove the span gas bag from the inlet.

Procedure:

1. Calibrate meter using the correct calibration procedure for the meter used.

2. Screening for VOCs will be accomplished by cutting the core and running the probe of the meter along the entire length of the core.

3. Take the highest reading from the meter or readout of the instrument.

4. Record the reading in the field logbook.

5. Recheck calibration with calibration gas after a minimum of every 10 samples and after the last sample.

Quality Control:

1. Calibration check results must be ±10 percent of the true value. If the result is outside of the ±10 percent, recalibrate the meter as specified above.

2. Duplicate samples are not analyzed since the headspace VOC readings will vary considerably as soil VOCs volatilize.

Interferences and Limitations:

Humid conditions will cause a negative bias to the reading. the photoionization detection principle used by the instruments will not detect all VOCs. The instrument is most sensitive to aromatic, alkene and alkyne VOCs.

APPENDIX C

Laboratory Analytical Standard Operating Procedures

Title: MERCURY IN SOLID OR SEMISOLID WASTE (MANUAL COLD-VAPOR TECHNIQUE) SW-846 METHOD 7471A/B

Approvals (Signature/Date):

Rodney Street — 7.23.10
Department Supervisor — Date

Michael H. Dunn — 7-20-10
Technical Director — Date

Jared Knierim — 7-23-10
Health & Safety Manager / Coordinator — Date

Eric S. Smith — 8/30/10
Quality Assurance Director — Date

Johnny A. Mitchell — 8.30.10
Laboratory Director — Date

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1.0 Scope and Application

1.1 Analyte, Matrices: This method is used for measuring total Mercury (organic and inorganic) in soils, sediments, bottom deposits, and sludge-type materials.

1.2 Reporting Limit (RL): The typical laboratory RL is 0.1 mg/kg.

1.3 If for any reason a part of this method cannot be followed, seek the guidance of the Department Supervisor or the Laboratory Technical Director. All abnormalities must be noted on the data or the benchsheet and in the Laboratory Information Management System (LIMS).

2.0 Summary of Method

This method is based on cold-vapor atomic absorption at the 253.7-nm wavelength. The Mercury is reduced to the elemental state and aerated from solution in a closed system. The Mercury vapor passes through a cell positioned in the light path of an atomic absorption spectrophotometer. Absorbance is measured as a function of Mercury concentration.

3.0 Definitions

See TestAmerica Nashville's Quality Assurance Manual Appendix 5 for laboratory definitions. Also, refer to Controlled Document QAF-45, TestAmerica Nashville Acronyms, Keywords, and Definitions.

4.0 Interferences

4.1 Potassium permanganate is added to eliminate possible interference from sulfide.

4.2 Copper has also been reported to interfere; however, Copper concentrations as high as 10 mg/kg had no effect on recovery of Mercury from spiked samples.

4.3 Samples high in chlorides require additional permanganate because, during the oxidation step, chlorides are converted to free chlorine, which also absorbs radiation of 253 nm. Care must therefore be taken to ensure that free chlorine is absent before the Mercury is reduced and swept into the cell. This may be accomplished by using an excess of hydroxylamine sulfate reagent or by dilution of the sample.

5.0 Safety

Employees must abide by the policies and procedures in the Corporate Safety Manual and this document. This procedure may involve hazardous material, operations and equipment. This method does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.1 Specific Safety Concerns or Requirements: Samples that contain high concentrations of carbonates or organic material or samples that are at elevated pH can react violently when acids are added.

5.2 Primary Materials Used: The following is a list of the materials used in this method, which have a serious or significant hazard rating. **Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

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Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Mercury	Oxidizer Corrosive Poison	0.1 mg/m^3 Ceiling (Mercury Compounds)	Extremely toxic. Causes irritation to the respiratory tract. Causes irritation. Symptoms include redness and pain. Can cause burns. Can cause sensitization. Can be absorbed through the skin with symptoms to parallel ingestion. Can affect the central nervous system. Causes irritation and burns to eyes. Symptoms include redness, pain, and blurred vision; can cause serious and permanent eye damage.
Nitric Acid	Corrosive Oxidizer Poison	2 ppm-TWA 4 ppm-STEL	Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison. Inhalation of vapors can cause breathing difficulties and lead to pneumonia and pulmonary edema, which can be fatal. Other symptoms can include coughing, choking, and irritation of the nose, throat, and respiratory tract. Can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and can cause damage to the eyes. Contact can cause severe burns and permanent eye damage.
Hydrochloric Acid	Corrosive Poison	5 ppm-Ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and can cause damage to the eyes. Contact can cause severe burns and permanent eye damage.
Potassium Permanganate	Oxidizer	5 mg/m^3 for Mn Compounds	Causes irritation to the respiratory tract. Symptoms can include coughing, shortness of breath. Dry crystals and concentrated solutions are caustic causing redness, pain, severe burns, brown stains in the contact area and possible hardening of outer skin layer. Diluted solutions are only mildly irritating to the skin. Eye contact with crystals (dusts) and concentrated solutions causes severe irritation, redness, and blurred vision and can cause severe damage, possibly permanent.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

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6.0 Equipment and Supplies

6.1 Instrumentation

- Leeman Mercury Analyzer PS-200 II, or equivalent. Instrument settings recommended by the particular manufacturer are followed.
- Data Acquisition, Leeman software.
- Air pump: Any peristaltic pump capable of delivering 1 L/minute air is used.
- Flowmeter, capable of measuring an air flow of 1 L/minute.
- Hot water bath or digestion block, adjustable and capable of maintaining a temperature of 92-98°C.

6.2 Supplies

- Volumetric flasks and pipets of suitable precision and accuracy (Class A).
- Teflon™ boiling chips (Mercury-free blank matrix).
- Polypropylene, certified, centrifuge tubes, 50 mL.

7.0 Reagents and Standards

7.1 Reagent water, analyte and interference-free, ASTM Type II or equivalent.

7.2 Hydrochloric acid, HCl, concentrated, commercial.

7.3 Nitric acid, HNO_3, concentrated, commercial.
7.4 Aqua regia: Prepare immediately before use by carefully adding **three** volumes of concentrated HCl to **one** volume of concentrated HNO_3.
7.5 Stannous chloride: Add 100 g stannous chloride to 100 mL HCl, dilute to 1000 mL with reagent water in a 1-L container.
7.6 Sodium chloride-hydroxylamine sulfate solution: Dissolve 120 g of sodium chloride and 120 g of hydroxylamine sulfate in reagent water and dilute to 1000 mL with reagent water in a 1-L container.
7.7 Potassium permanganate, Mercury-free, 5% solution (w/v): Dissolve 50 g of potassium permanganate in 1000 mL of reagent water in a 1-L container.
7.8 Mercury stock solution: Certified commercial source (1000 µg/mL).

- **Mercury intermediate working standard**: Make dilution of the stock Mercury solution to obtain a working standard containing 1.0 µg/mL. Dilute 0.05 mL stock to 50.0 mL with 1% HNO_3 (10 mL concentrated acid to 1 L reagent water) in a Class A, volumetric flask. Prepare monthly.

7.7 See SOP Reagent and Standard Purchase, Preparation, Control, and Documentation / NV08-214 for shelf-life and storage requirements for reagents and standards.

8.0 Sample Collection, Preservation, Shipment and Storage

Matrix	Sample Container	Min. Sample Size	Preservation	Holding Time	Reference
Solid	HDPE or Glass	1-10 g	Less than or equal to 6 degrees C.	28 days from collection	SW-846 Chapter 2

9.0 Quality Control

Refer to the quality control section of TestAmerica Nashville's QA Manual for specific quality control (QC) policies. The laboratory maintains a formal quality assurance program and records to document the quality of the data generated.

9.1 Sample QC

The following quality control samples are prepared with **each batch of no more than 20 samples, except for samples from OK and WY which are 10 samples per batch.**			
Quality Controls	**Frequency**	**Control Limit**	**Corrective Action**
Method Blank	1 each batch	< RL	Correct problem, re-prep and analyze the MB, all samples, and QC in the affected analytical batch.
Laboratory Control Sample[1] (LCS), second source	1[1] each batch	80-120% recovery	Correct problem, then re-prep and re-analyze the LCS, all samples, and QC in the affected analytical batch. If high and target is ND, OK to report.
Matrix Spike (MS), second source	1 each batch	7471A: 75-125% recovery 7471B: 80-120% recovery	Report and qualify

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The following quality control samples are prepared with **each batch of no more than 20 samples, except for samples from OK and WY which are 10 samples per batch.**			
Quality Controls	**Frequency**	**Control Limit**	**Corrective Action**
Matrix Spike Duplicate (MSD), second source	1 each batch	7471A: 75-125% recovery 7471B: 80-120% recovery RPD< 20%	Report and qualify
Dilution Test	Diluted samples	5X sample result must be 90-110% of the undiluted sample result.	Run post-digestion spike
Post-digestion Spike	If MS/MSD fails	80-120% recovery	Dilute and re-analyze.

[1]For AZ, TX, WV samples, a LCS duplicate is required.

- **Method blank:** The method blank starts with the addition of 50 mL reagent water and aqua regia to 0.6 g boiling chips.
- A **Laboratory Control Sample (LCS):** To evaluate the ability of analyzing a clean matrix of known concentration, prepare one LCS per batch exactly as client samples, as follows:
  - Add 0.100 mL of the intermediate secondary working solution to 0.6 gram Teflon™-boiling chips. Dilute to 100 mL in a Class A volumetric flask with reagent water for a 1.0 µg/L final concentration in the digestate.
- **Matrix Spike/Matrix Spike Duplicate Pair:** To evaluate the effect of the matrix on the analysis, analyze two, identical, 0.6 gram aliquots of a client sample.
  - Spike each aliquot with 0.100 mL of the intermediate secondary standard. Digest and dilute to 100 mL in a Class A volumetric flask with reagent water for a 1.0 µg/L final concentration in the digestate
  - If the recovery for the MS/MSD cannot be determined, due to the high concentration (about four times greater than the spike level) of the sample used for spiking, then run a dilution test.
  - If the MS/MSD % recovery is not met, perform a post-digestion spike.
- **Dilution Test:** If there is a sample in the batch greater than 5.0 µg/kg, dilute the sample by a minimum of five-fold and reanalyze. If results do not agree, run a post-digestion spike.
- **Post-digestion Spike:** If the post-digestion spike does not meet the QC criteria, dilute the original sample and re-run.

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9.2 Instrument QC

Quality Controls	Frequency	Control Limit	Corrective Action
Initial Calibration Verification Sample (ICV), second source	Immediately after calibration	90-110% recovery	Correct problem, then repeat initial calibration.
Initial Calibration Blank (ICB)	Immediately after ICV	< MDL	Correct problem then re-digest and reanalyze calibration and entire digestion batch.
Continuing Calibration Verification Sample (CCV), primary source	Every 10 samples	7471A: 90-110% recovery 7471B: 80-120% recovery	Correct problem; repeat all QC and samples since last successful CCV. If high and target is ND, OK to report.
Continuing Calibration Blank (CCB)	Following each CCV	< RL	Correct problem, repeat all QC and samples associated with that CCB.

Quality Controls	Frequency	Control Limit	Corrective Action
Instrument Detection Limits (IDL)	Quarterly	±3 times standard deviation of the average response of blank.	Compare with MDL and re-run if IDL > MDL. If the IDL is still > MDL, elevate the MDL to the IDL. Update LIMS.
Report Limit Verification (RLV)	Daily	Required percent recovery is ±30%.	Perform maintenance, recalibrate.
MDL Verification	Minimum annually	Detect	Re-evaluate MDL standard used and MDL; see Technical Director.

- **Initial Calibration Verification (ICV) and Initial Calibration Blank (ICB):** The laboratory analyzes an ICV and an ICB immediately following daily calibration.
  - For the ICV, add 250 µL of the secondary intermediate standard to 100 mL of reagent water in a Class A volumetric flask for a 2.5 µg/L final concentration.
  - The ICB contains the same acid and reagent water as the calibration standards; it is not digested.
- **Instrument Detection Limit (IDL):** - On a quarterly basis, analyze 10 CCBs consecutively. Calculate the average response and standard deviation. The IDL is ± 3SD of the average response. Compare to the MDL.
- **Continuing Calibration Verification (CCV and CCB):** Analyze after every 10th sample and at the end of the analytical sequence.
  - Prepare the CCV at 2.0 µg/L by adding 200 µL of the primary Mercury standard to 100 mL reagent water in a Class A volumetric flask. Digest the CCV in the same manner as the samples.
  - The CCB contains the same acid and reagent water as the calibration standards; it is digested.
- **Report Limit Verification (RLV):** Daily analyze a standard at the RL standard, prepared like the samples. If the percent recovery falls outside the control limits, the RLV must be re-analyzed immediately. If it fails again, correct problem, re-calibrate. The RLV may also be used as the MDL verification (1-3 X MDL) and establishes the report limit.

10.0 Procedure

10.1 Sample Preparation

Matrix	Sample Size
Solid	0.6 gram

1	Weigh a 0.6-g portion of a well homogenized sample (three 0.2 g portions) and place in the bottom of a clean, certified, polypropylene tube. See SOP Sample Homogenization, Subsampling, and Compositing / NV08-229.
2	Prepare the spiked QC samples as described in Section 9.1.
3	Add 5.0 mL of reagent water and 5.0 mL of aqua regia to all samples and QC.
4	Heat 2 minutes in a hot block at 95 ±3°C. Record time and temperature. Vary the location of the thermometer weekly and record the position.
5	Cool; then add 10.0 mL reagent water and 15.0 mL potassium permanganate solution to each sample bottle.
6	Mix thoroughly, cap, and place in the hot block for 30 minutes at 95 ± 3°C. Record.
7	Cool and add 6.0 mL of sodium chloride-hydroxylamine sulfate to reduce the excess

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	permanganate.
8	Dilute to 100 mL in a Class A volumetric flask. Transfer an aliquot to a centrifuge tube for storage.

10.2 Calibration: Refer to SOP Selection of Calibration Points / CA-P-T-002. See Section 11 for equations. Calculations are performed by vendor software and LIMS.

1	Start-up the instrument computer, using "WinHg runner," the software: Go to the control tab and turn the pump on at 5 mL/minute. Turn lamp and gas on. The instrument turns on and goes through a warm-up period. Because of instrument variation refer to the manufacturer's recommended operating conditions when using this method. Allow the instrument to proceed through a warm-up period before beginning the calibration sequence.
2	**Each day of analysis**, prepare a fresh set of the following calibration standards in clean, certified, centrifuge tubes:

µL of 1.0 µg/mL Primary Standard to 100 mL Reagent Water	Calibration Standard Concentration (µg/L)
0	0
20	0.2
50	0.5
100	1.0
200	2.0
250	2.5
500	5.0

	Note: The lowest non-zero calibration standard is at or below the RL.
3	Add 5.0 mL reagent water to each.
4	Add 5.0 mL aqua regia and heat 2 minutes in a water bath at 95 ±3°C. Record. Allow the sample to cool.
5	Add 10.0 mL reagent water and 15.0 mL $KMnO_4$ solution to each tube, and return to the water bath for 30 minutes at 95 ± 3°C. Record. Cool.
6	Add 6.0 mL sodium chloride-hydroxylamine sulfate solution to reduce the excess permanganate.
7	Dilute to 100 mL reagent water in a Class A volumetric flask.
8	Place the appropriate standards for the method selected in the standard rack. Click on the standard tab.
9	Depress standard buttons for standards #1, #2, #3, #4, #5, #6, and #7.
10	Depress Rep 1 and Rep 2 buttons, then click standard Auto. The instrument will begin calibration.
11	To view first-order linear regression curve, click database button and the cal curve tab. The new curve is displayed. If its correlation coefficient, r, is greater than or equal to 0.995 ($r^2 \geq 0.99$), the new curve is stored. Higher order, nonlinear models are not allowed.
12	If the correlation coefficient requirement is not met, prepare the calibration standards again and repeat calibration.
13	When calibrating replicates > RL, RPD must be <10. For the blank and RL standard replicates, RPD must be <30. If the RPD fails for a concentration level, run two new replicates.

10.3 Sample Analysis

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1	Replenish the stannous chloride reservoir for automated addition of reagent.
2	Click rack editor button open appropriate rack file (1 or 2), then enter sample ID into appropriate cup position.
3	Save information before returning to "WinHg runner."
4	Return to " WinHg runner" and click sample tab.
5	Choose rack number to be run, start cup number and end cup number.
6	To begin click run auto button, the instrument will begin the sequence. Print the raw absorbance and concentration for each sample and QA/QC.
7	To shutdown the procedure, return to the control tab. Turn gas and pump off and loosen tubes. Turn off the power to the lamp if the instrument will not be used for 24 hours or longer.

10.4 Example Analysis Queue / Sequence

1	ICAL (daily)
2	ICV (daily)
3	ICB (daily)
4	RLV (daily)
5	Method Blank
6	LCS
7	Matrix Spike
8	Matrix Spike Duplicate
9	Samples 1-10
10	CCV
11	CCB
12	Samples 11-20
13	CCV
14	CCB

11.0 Calculations / Data Reduction

11.1 Accuracy

$$\textbf{LCS \% Recovery} = \frac{\text{Measured concentration}}{\text{Known concentration}} \times 100$$

11.2 Precision (RPD)

$$\textbf{RPD} = \frac{\text{Absolute value (orig. sample value - dup. sample value)} \times 100}{\text{(Orig. sample value + dup. sample value)/2}}$$

11.3 Linear calibration using a least squares regression

Perform a first-order linear regression of the instrument response versus the concentration of the standards. Non-linear equations are not allowed for this analysis. Make certain that the instrument response is treated as the dependent variable (y) and the concentration as the independent variable (x). This is a statistical requirement and is not simply a graphical convention.

The regression produces the slope and intercept terms for a linear equation in the form:

$y = ax + b$

y = instrument response (peak absorbance)

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a = slope of the line
x = concentration of the calibration standard
b = the intercept

The analyst must not force the line through the origin, but have the intercept calculated from the data points. The use of a linear regression is not used as a rationale for reporting results below the calibration range demonstrated by the analysis of the standards. The regression calculation generates a correlation coefficient (r) that is a measure of the "goodness of fit" of the regression line to the data. A value of 1.00 indicates a perfect fit. In order to be used for quantitative purposes, r must be greater than or equal to 0.995 or $r^2 \geq 0.99$.

11.4 Coefficient of Determination

$$r^2 = \frac{\left(\sum xy\right)^2}{\sum x^2 \sum y^2}$$

Correlation Coefficient

$$r = \frac{\left(\sum xy\right)}{\sqrt{\sum x^2 \sum y^2}}$$

y = Response
x = Concentration

11.5 Record and print metal concentrations directly from the instrument's concentration read-out. All dilution or concentration factors must be taken into account.

Concentration (µg/g) = $\frac{\text{(µg/L from instrument) (dilution factor)}}{\text{grams of sample}}$

If no dilution, the dilution factor is 1.

12.0 Method Performance

12.1 Method Detection Limit Study (MDL): The method detection limit (MDL) is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. The MDL is determined according to the laboratory's MDL procedure in SOP Determination of Method Detection Limits / NV08-202. MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix, and may not be achievable in all environmental matrices. The laboratory maintains MDL studies for analyses performed; these are verified at least annually unless the method requires a greater frequency.

12.2 Demonstration of Capability: The laboratory demonstrates initial proficiency by generating data of acceptable accuracy and precision for target analyses in a clean matrix. The laboratory also repeats the operation whenever new staff is trained or significant changes in instrumentation are made and on an annual basis thereafter. See the training section of TestAmerica Nashville's QA Manual and SOP Training / NV08-199 for information on how to accomplish this demonstration.

12.3 Training Requirements: Demonstration of Capability is performed initially when learning the method and annually thereafter. Four Laboratory Control Samples resulting in an average % recovery within the control limits and a precision less than the quality control maximum are required for each matrix.

12.4 Proficiency Testing Studies: The laboratory participates in formal proficiency testing (PT) studies, where solutions of unknown concentrations are analyzed and the performance of all participants is compared. See the QA department for the results of recent PT studies.

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13.0 Pollution Control

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i. e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention."

14.0 Waste Management

14.1 Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are managed, stored, and disposed of in accordance with all federal and state laws and regulations. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to the QA Manual and SOP Waste Disposal / NV10-83.

14.2 Wastestreams Produced by the Method:

- Digestates are disposed of in the Mercury waste drum in the waste disposal area.

15.0 References / Cross-References

15.1 SW-846 Methods 7471A, Update III, September 1994, and 7471B, Revision 2, February 2007.

15.2 TestAmerica Nashville's Quality Assurance Manual.

15.3 TestAmerica Nashville's Control Limits Manual.

15.4 Corporate Environmental Health and Safety Manual (CW-E-M-001)

15.5 SOPs: Waste Disposal / NV10-83, Training Procedures for Environmental Technical Staff / NV08-199, Balance Calibration / NV08-213, Determination of Method Detection Limits / NV08-202, Sample Homogenization, Subsampling, and Compositing / NV08-229, Selection of Calibration Points / CA-P-T-002.

15.6 Controlled Document: QAF-45, TestAmerica Nashville – Acronyms, Keywords, and Definitions.

16.0 Method Modifications

None.

17.0 Attachments

None.

18.0 Revision History

- Revision 10, dated 30 September 2008
  - Integration of TestAmerica and STL operations.
  - Update to 7471B.
- Revision 11, dated 25 September 2009
  - Addition of OH VAP requirements.
- Revision 12, dated 31 August 2010
  - Addition of QAF-45, Section 14.2, and SOP Selection of Calibration Points / CA-P-T-002.
  - Distinguish QC requirements for 7471A and 7471B.
  - Refer to hot block rather than water bath. Add instruction to vary the location of the thermometer in the hot block weekly and record the thermometer's position.
  - Delete redundant information and add standardized text.
  - Add instruction to print the raw absorbance and concentration information.

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THE LEADER IN ENVIRONMENTAL TESTING

Nashville Standard Operating Procedure (SOP) Change Form

SOP Number/Revision No.: **7470 / NV06-41.11a** Effective Date: 11/30/10

Last Mod. Date: **12/30/09**

SOP Title: **Method 7470A: Mercury in Liquid Waste (Manual Cold Vapor Technique)**

Affected SOP Section Number(s): Section 10.4, Example Analysis Queue / Sequence*

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ISSUED TO: QA Server, QA, 06

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Revision Number with Mod ID: **11b**

The following SOP change is in effect as of the stated date. This form will remain attached to the referenced SOP until such a time that the SOP is updated, approved, and redistributed, at which time it will become part of the historical SOP record. ***Append this form to the front of the SOP copy.***

1. Reason for SOP Change:

☐ Typographical Corrections (Non-Technical) – Re-Training Not Required.

☐ Typographical Corrections (Technical – Define) – Analyst acknowledgement of corrections is required.

☐ Procedural Changes (Define Below) – Re-Training Required.

■ Other: Audit recommendation.

2. Summary of Procedure Change

Section 10.4, Example Analysis Queue / Sequence*: Change "ICAL" to "Initial Calibration."

[signature]	11-30-10	[signature]	11-30-10
Supervisor Approval	Date	Technical Director Approval	Date
[signature]	11/30/10	[signature]	11-30-10
Quality Assurance Approval	Date	Laboratory Director Approval	Date

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

Nashville
Standard Operating Procedure (SOP)
Change Form

SOP Number/Revision No.: **7470 / NV06-41.11** Effective Date: **12/30/09**
Last Mod. Date: 9/25/09

SOP Title: **Method 7470A: Mercury in Liquid Waste (Manual Cold Vapor Technique)**

Affected SOP Section Number(s): Calibration Section

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ISSUED TO: QA Server, QA, 06

Revision Number with Mod ID: **11a**

The following SOP change is in effect as of the stated date. This form will remain attached to the referenced SOP until such a time that the SOP is updated, approved, and redistributed, at which time it will become part of the historical SOP record. ***Append this form to the front of the SOP copy.***

1. Reason for SOP Change:

☐ Typographical Corrections (Non-Technical) – Re-Training Not Required.

☐ Typographical Corrections (Technical – Define) – Analyst acknowledgement of corrections is required.

■ Procedural Changes (Define Below) – Re-Training Required.

☐ Other

2. Summary of Procedure Change

Calibration Section, add the following guidance:

When calibrating replicates > RL, RPD must be <10. For the blank and RL standard replicates, RPD must be <30. If the RPD fails for a concentration level, run two new replicates.

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[signature]	11-12-09	[signature]	11/12/09
Supervisor Approval	Date	Technical Director Approval	Date
[signature]	11/30/09	[signature]	12-2-09
Quality Assurance Approval	Date	Laboratory Director Approval	Date

Title: MERCURY IN LIQUID WASTE (MANUAL COLD VAPOR TECHNIQUE) Method 7470A

Approvals (Signature/Date):

Rodney Street — 9-21-09
Department Supervisor — Date

Michael H. Dunn — 9-18-09
Technical Director — Date

Jared Knierim — 9-22-09
Health & Safety Manager / Coordinator — Date

Eric S. Smith — 9/17/09
Quality Assurance Director — Date

Johnny A. Mitchell — 9.24.09
Laboratory Director — Date

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1.0 Scope and Application

1.1 Analyte, Matrices: This method uses cold-vapor atomic absorption for determining the concentration of mercury (CAS # 7439-97-6) in mobility-procedure extracts, aqueous wastes, and ground waters.

1.2 Reporting Limits: The typical detection limit for this method is 0.0002 mg/L.

1.3 The intent of the words "must," "shall," "should," or "will" renders the statement a mandatory requirement of this SOP.

1.4 If for any reason a part of this SOP cannot be followed, seek the guidance of the Department Supervisor or the Laboratory Technical Director. All abnormalities must be noted on the data or the benchsheet, and in LIMS. See the corrective action policies in the QA Manual.

2.0 Summary of Method

This method is a cold-vapor atomic absorption technique and is based on the absorption of radiation at 253.7-nm, by mercury vapor. The mercury is reduced to the elemental state and aerated from solution in a closed system. The mercury vapor passes through a cell positioned in the light path of an atomic absorption spectrophotometer. Absorbance is measured as a function of mercury concentration.

3.0 Definitions

See TestAmerica Nashville's QA Manual Appendix 5 for other laboratory definitions.

4.0 Interferences

4.1 Potassium permanganate is added to eliminate possible interference from sulfide.

4.2 Copper has also been reported to interfere; however, copper concentrations as high as 10 mg/L had no effect on recovery of mercury from spiked samples.

4.3 Seawaters, brines, and industrial effluents high in chlorides **require** additional permanganate because, during the oxidation step, chlorides are converted to free chlorine, which also absorbs radiation of 253.7 nm. Care must therefore be taken to ensure that free chlorine is absent before the mercury is reduced and swept into the cell. This is accomplished by using an excess of hydroxylamine sulfate reagent.

5.0 Safety

Employees must abide by the policies and procedures in the Corporate Safety Manual and this document.

This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.1 Specific Safety Concerns or Requirements: Perform the digestion in an operational fume hood.

5.2 Primary Materials Used: The following is a list of the materials used in this method, which have a serious or significant hazard rating. **Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

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Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Mercury	Oxidizer Corrosive Poison	0.1 mg/m^3 Ceiling (Mercury Com-pounds)	Extremely toxic. Causes irritation to the respiratory tract. Causes irritation. Symptoms include redness and pain. Can cause burns. Can cause sensitization. Can be absorbed through the skin with symptoms to parallel ingestion. Can affect the central nervous system. Causes irritation and burns to eyes. Symptoms include redness, pain, and blurred vision; can cause serious and permanent eye damage.
Nitric Acid	Corrosive Oxidizer Poison	2 ppm-TWA 4 ppm-STEL	Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison. Inhalation of vapors can cause breathing difficulties and lead to pneumonia and pulmonary edema, which can be fatal. Other symptoms can include coughing, choking, and irritation of the nose, throat, and respiratory tract. Can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and can cause damage to the eyes. Contact can cause severe burns and permanent eye damage.
Hydro-chloric Acid	Corrosive Poison	5 ppm-Ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and can cause damage to the eyes. Contact can cause severe burns and permanent eye damage.
Potas-sium Perman-ganate	Oxidizer	5 mg/m^3 for Mn Com-pounds	Causes irritation to the respiratory tract. Symptoms can include coughing, shortness of breath. Dry crystals and concentrated solutions are caustic causing redness, pain, severe burns, brown stains in the contact area and possible hardening of outer skin layer. Diluted solutions are only mildly irritating to the skin. Eye contact with crystals (dusts) and concentrated solutions causes severe irritation, redness, and blurred vision and can cause severe damage, possibly permanent.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

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6.0 Equipment and Supplies

6.1 Instrumentation

- Leeman Mercury Cold Vapor Analyzer Hydra AA: Instrument settings recommended by the particular manufacturer are followed.
- Recorder: Data acquisition by PC running Leeman software.
- Air pump: Any peristaltic pump capable of delivering 1 liter air/minute is used.
- Flowmeter: Capable of measuring an air flow of 1 liter/minute.
- Hot water bath or digestion block. - Adjustable and capable of maintaining a temperature of 90-95°C.

6.2 Supplies

- Volumetric flasks and pipettes of suitable precision and accuracy (Class A).
- Certified, centrifuge tubes, 50-mL.

7.0 Reagents and Standards

7.1 Reagent water, analyte-free, ASTM Type II or equivalent.

7.2 Hydrochloric acid, HCl, concentrated, commercial.
7.3 Nitric acid (HNO_3), concentrated: Reagent grade of low mercury content. If a high reagent blank is obtained, it may be necessary to change lots of the nitric acid.
7.4 Stannous chloride: Add 100 g stannous chloride to 100 mL HCl, dilute to 1000 mL with reagent water in a 1-L container.
7.5 Sodium chloride-hydroxylamine sulfate solution: Dissolve 120 g of sodium chloride and 120 g of hydroxylamine sulfate in reagent water and dilute to 1000 mL with reagent water in a 1-L container. (Hydroxylamine hydrochloride may be used in place of hydroxylamine sulfate.)
7.6 Potassium permanganate, mercury-free, 5% solution (w/v): Dissolve 50 g of potassium permanganate in 1000 mL of reagent water in a 1-L container.
7.7 Potassium persulfate, 5% solution (w/v): Dissolve 50 g of potassium persulfate in 1000 mL of reagent water in a 1-L container.
7.8 Stock mercury solution: Stock solutions are purchased at 1000 µg/mL from CPI and Ultra Scientific. Use caution in handling mercury solutions.
7.9 Mercury intermediate working standard: Make dilution of the stock mercury solution to obtain a working standard containing 1.0 µg/mL. Dilute 0.05 mL 1000 µg/mL stock to 50.0 mL with 1% HNO_3 in a Class A, volumetric flask. Prepare monthly.
7.10 Initial Calibration Verification/Laboratory Control Sample (ICV/LCS): Prepare from a **different** source than the calibration standards.
7.11 See SOP Reagent and Standard Purchase, Preparation, Control, and Documentation / NV08-214 for shelf-life and storage requirements for reagents and standards.

8.0 Sample Collection, Preservation, Shipment and Storage

Matrix	Sample Container	Min. Sample Size	Preservation	Holding Time	Reference
Water	HDPE or Glass	30.0 mL	HNO_3 to pH ≤ 2	28 days from collection	SW846 Section 2.0

Temperature preservation is not used. Certified cleaned containers are used, if the containers are supplied by TestAmerica Nashville.

9.0 Quality Control

Refer to the quality control section of TestAmerica Nashville's QA Manual for quality control (QC) policies. The laboratory maintains a formal quality assurance program and records to document the quality of the data generated.

9.1 Sample QC

The following quality control samples are prepared with **each batch of no more than 20 samples, except for samples from OK and WY which require batches of no more than 10 samples.**			
Quality Controls	**Frequency**	**Acceptance Limits**	**Corrective Action**
Method Blank (MB)	1 per batch	< ½ RL	Correct problem, then re-prep and analyze method blank, all samples, and QC processed with the contaminated blank.

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The following quality control samples are prepared with **each batch of no more than 20 samples, except for samples from OK and WY which require batches of no more than 10 samples.**			
Quality Controls	**Frequency**	**Acceptance Limits**	**Corrective Action**
Laboratory Control Sample[1] (LCS), second source	1[1] per batch	80-120% recovery	Correct problem, then re-prep and analyze the LCS, all samples, and QC in the affected analytical batch. If high and sample is ND, it is OK to report.
Matrix Spike	1 per batch	75-125% recovery	Report and qualify
Matrix Spike Duplicate	1 per batch	75-125% recovery, < 20% RPD	Report and qualify
Dilution Test	1 per batch	5X dilution sample result must be 90-110% of the undiluted sample result.	Perform post-digestion spike.
Post-digestion Spike	If results of dilution test do not agree.	80-120%	Dilute and reanalyze

1 All AZ, TX, and WV samples require a LCS duplicate in each batch.

- **Method or preparation blank:** The laboratory prepares and analyzes at least one blank with each batch of the same matrix. Blank data are used to assess contamination from the laboratory environment. Blank values that exceed ½ the RL indicate laboratory or reagent contamination are suspect.
- A **Laboratory Control Sample (LCS):** To evaluate the ability of analyzing a clean matrix of known concentration, prepare one LCS per batch exactly as client samples, and compare the % recovery to the control limits.
  - Add 30 µL of 1.0 µg/mL standard to 30.0 mL reagent water for the LCS in a certified, centrifuge tube for a final concentration of 1.0 µg/L.
- A **Matrix Spike/Matrix Spike Duplicate (MS/MSD)** are analyzed every batch. The MS/MSD limits are 75-125% recovery, ≤ 20% RPD. If the recovery for the MS/MSD cannot be determined, due to the high concentration (4 times greater than the spike level) of the sample used for spiking, then run a dilution test.
  - The spike solution is made by adding 30 µL of 1.0 µg/mL standard to 30.0 mL reagent water for the MS/MSD in a certified, centrifuge tube for a final concentration of 1.0 µg/L.
- **Dilution Test** - Dilute the sample by a minimum of five fold and re-analyze. Agreement within 10% between the concentration for the undiluted sample and five times the concentration for the diluted sample indicates the absence of interferences. If the results do not agree, run a post-digestion spike.
- **Post-Digestion Spike** – If the post-digestion spike is not within 80-120%, dilute the original sample and re-run.

9.2 Instrument QC:

Quality Controls	Frequency	Control Limit	Corrective Action
Initial Calibration Verification Sample (ICV), second source	Immediately after calibration	90-110%	Correct problem, then repeat initial calibration.
Initial Calibration Blank	Beginning of each batch	< MDL	Correct problem, then re-digest and re-analyze calibration and entire

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Quality Controls	Frequency	Control Limit	Corrective Action
			digestion batch.
CRI / Report Limit Verification (RLV)/MDLV	Beginning of each batch	0.14-0.26 µg/L, ± 30% true	Correct problem, rerun, recalibrate
Continuing Calibration Verification Sample (CCV)	Every 10 samples and end of batch	90-110%	If CCV falls outside of range low rerun once and if still out of range, correct problem, recalibrate and rerun all affected samples. If high and sample is ND, OK to report.
Continuing Calibration Blank	Following the CCV	< ½ RL	Rerun affected samples.
Instrument Detection Limits (IDL)	Quarterly	±3 times standard deviation of the average response of blanks.	Update MDL/IDL comparison. If IDL > MDL, correct problem and rerun IDL.
MDL Verification	Minimum annually	Detect	Re-evaluate MDL standard used and MDL; see Technical Director.

- **Initial Calibration Verification (ICV)** and **Initial Calibration Blank (ICB):** A verification standard made from a **different** source than the calibration standards is run immediately after calibration. The standard must fall within 10% of true value or recalibrate.
  - Prepare ICV by adding 75 µL of the 1.0 µg/mL second-source standard to 30.0 mL acidified reagent water in a certified, centrifuge tube for a final concentration of 2.5 µg/L. The ICV must agree within ± 10% of the true value.
  - The ICB contains the same acid and reagent water as the calibration standards; they are not digested. The concentration must be less than the MDL.
- **Continuing Calibration Verification** (CCV) and **Continuing Calibration Blank (CCB)**: Every 10 samples and at the end of the sequence, a mid-level standard (2.0 µg/L) is analyzed and must fall within 10% of the true value. If the CCV is outside of range, rerun once and, if still out of range, correct problem, recalibrate and rerun all affected samples.
  - Prepare CCV by adding 60 µL of the 1.0 µg/mL primary source standard to 30.0 mL acidified reagent water in a certified, centrifuge tube for a final concentration of 2.0 µg/L.
  - The CCB contains the same acid and reagent water as the calibration standards; they are not digested. The concentration must be less than ½ the RL.
- **Instrument Detection Limits (IDL):** On a quarterly basis, analyze 10 CCBs consecutively. Calculate the average response and standard deviation. The IDL is ± 3SD of the average response. Update LIMS with new information. IDL must be ≤ MDL.
- **CRI** (low verification), **Report Limit Verification** (RLV): **Method Detection Limit Verification** (MDLV): Daily, analyze a low calibration verification at 0.2 µg/L at the beginning of the analytical sequence. If the percent recovery falls outside the limits (± 30%), the CRI must be re-analyzed immediately. If it fails again, correct the problem, re-calibrate. The CRI/RLV is also used as the MDL verification and establishes the report limit. The MDLV criterion is met when the standard is detected.
  - Prepare by diluting the 2.0 µg/L CCV standard by 10x (3.0 mL to 30.0 mL). The acceptance range is 0.14 µg/L to 0.26 µg/L.

10.0 Procedure

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10.1 Sample Preparation

Matrix	Sample Size
Water	30 mL of sample

1	Transfer 30.0 mL of sample into a 50-mL, certified, centrifuge tube. For the LCS, add 30.0 mL reagent water to a 50-mL, certified, centrifuge tube.
2	Add 1.5 mL of H_2SO_4 and 0.75 mL of concentrated HNO_3, mixing after each addition.
3	Add 4.5 mL of 5% potassium permanganate solution to each sample bottle. Seawater and brine may need additional amount added. Ensure that equal amounts of permanganate are added to standards and blanks. Shake and add additional portions of potassium permanganate solution, if necessary, until the purple color persists for at least 15 minutes.
4	Add 2.4 mL of potassium persulfate to each bottle and heat for 120 ± 10 minutes in a water bath maintained at 95 ± 2°C. Record start and stop time and temperature.
5	Cool and add 1.8 mL of sodium chloride hydroxylamine sulfate to reduce the excess permanganate. Additional hydroxylamine solution may be needed to complete de-colorization. Place in autosampler and start analysis.
6	Spike the LCS and MS/MSD.
7	For the method blank, use 30.0 mL of reagent water.

10.2 Calibration

1	Start-up the instrument computer, using "WinHg runner," the software: Go to the control tab and turn the pump on at 5 mL/minute. Turn lamp and gas on. The instrument turns on and go through a warm up period. Because of instrument variation refer to the manufacturer's recommended operating conditions when using this method. Allow the instrument to proceed through a warm-up period before beginning the calibration sequence.
2	Standard preparation: **Daily**, prepare calibration standards from 1.0 µg/mL working standard in 30.0 mL of 1% HNO_3.

mL Standard/30.0 mL final volume	Final Concentration, µg/L
0.0	0.0
0.006	0.2
0.015	0.5
0.030	1.0
0.060	2.0
0.075	2.5
0.150	5.0

3	Transfer the standard into a 50 mL polypropylene centrifuge tube.
4	Mix thoroughly and add 1.5 mL of concentrated H_2SO_4 and 0.75 mL of concentrated HNO_3 to each bottle.
5	Add 4.5 mL of $KMnO_4$ solution to each bottle and allow to stand at least 15 minutes.
6	Add 2.4 mL of potassium persulfate to each bottle and heat for 2 hours in a water bath maintained at 95 ± 2°C. (Record start and stop time, and temperature.)
7	Cool and add 1.8 mL of sodium chloride-hydroxylamine sulfate solution to reduce the excess permanganate. Place in autosampler and start analysis.
8	After computer start-up (see **Sample Analysis** below), put the appropriate standards for the method selected in the standard rack. Click on standard tab.
9	Depress standard buttons for standards 1, 2, 3, 4, 5, and 6.

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10	Depress Rep 1 and Rep 2 buttons, then click standard Auto. The instrument begins calibration, using a linear calibration curve model. **Non-linear models are not allowed.**
10	To view first order linear regression curve, click data base button and the cal curve tab. The new curve is displayed. If its correlation coefficient $r \geq 0.995$ (or $r^2 \geq 0.99$), then check the accepted box and the new curve is stored. See Section 11.0 for equations.

10.3 Sample Analysis

1	Replenish the stannous chloride reservoir for automated addition of reagent.
2	Running samples: • Click rack editor button to open appropriate rack file (1 or 2), then enter sample ID into appropriate cup position. • Save information before returning to WinHg runner. • Return to runner and click sample tab. • Choose rack number to be run, start cup number and end cup number. • To begin, click run auto button; the instrument begins the sequence. Spiked samples and check standards are analyzed.
3	Shut-down procedure: • Return to control tab. Turn gas and pump off, and loosen tubes. • Turn off the power to the lamp if the instrument is not used for 24 hours or longer.

10.4 Example Analysis Queue / Sequence*

	Sample ID
1	ICAL (daily)
2	ICV (daily)
3	ICB (daily)
4	CRI/RLV (daily)
5	Method Blank
6	LCS
7	Matrix Spike
8	Matrix Spike Duplicate
9	Samples 1-10
10	CCV
11	CCB
12	Samples 11-20
13	CCV
14	CCB

May be up to 20 samples

11.0 Calculations / Data Reduction

11.1 Accuracy

$$\textbf{LCS \% Recovery} = \frac{\text{measured concentration}}{\text{known concentration}} \times 100$$

11.2 Precision (RPD)

$$\textbf{RPD} = \frac{\text{Absolute value (orig. sample value - dup. sample value)}}{} \times 100$$

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(orig. sample value + dup. sample value)/2

11.3 Linear calibration using a least squares regression

Performing a linear regression of the instrument response versus the concentration of the standards. Non-linear equations are not allowed for this analysis. Make certain that the instrument response is treated as the dependent variable (y) and the concentration as the independent variable (x). This is a statistical requirement and is not simply a graphical convention.

The regression produces the slope and intercept terms for a linear equation in the form:

$$y = ax + b$$

y = instrument response (peak area)
a = slope of the line
x = concentration of the calibration standard
b = the intercept

Do not force the line through the origin, but have the intercept calculated from the data points. The use of a linear regression is not used as a rationale form reporting results below the lowest calibration standard. The regression calculation generates a correlation coefficient (r) that is a measure of the "goodness of fit" of the regression line to the data. A value of 1.00 indicates a perfect fit. In order to be used for quantitative purposes, r must be greater than or equal to 0.995 or $r^2 \geq 0.99$.

11.4 Coefficient of Determination

$$r^2 = \frac{\left(\sum xy\right)^2}{\sum x^2 \sum y^2}$$

Correlation Coefficient

$$r = \frac{\left(\sum xy\right)}{\sqrt{\sum x^2 \sum y^2}}$$

where
y = Response or Response ratio
x = Concentration

11.5 Sample Concentrations: Record metal concentrations directly from the instrument's concentration read-out. All dilution or concentration factors must be taken into account.

Concentration (µg/L) = (µg/L from instrument) (dilution factor)

Dilution factor = 1 if there is no dilution.

12.0 Method Performance

12.1 Method Detection Limit Study (MDL): The method detection limit (MDL) is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. The MDL is determined according to the laboratory's MDL procedure in SOP Determination of Method Detection Limits / NV08-202. MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix, and may not be achievable in all environmental matrices. The laboratory maintains MDL studies for analyses

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performed; these are verified at least annually unless method requirements require a greater frequency.

12.2 Demonstration of Capability: The laboratory demonstrates initial proficiency by generating data of acceptable accuracy and precision for target analyses in a clean matrix. The laboratory also repeats the operation whenever new staff is trained or significant changes in instrumentation are made and on an annual basis thereafter. See the training section of TestAmerica Nashville's QA Manual and SOP Training / NV08-199 for information on how to accomplish this demonstration.

12.3 Training Requirements: Demonstration of Capability is performed initially when learning the method and annually thereafter. Four Laboratory Control Samples resulting in an average % recovery within the control limits and a precision less than the quality control maximum are required for each matrix.

12.4 Proficiency Testing Studies: The laboratory participates in formal proficiency testing (PT) studies, where solutions of unknown concentrations are analyzed and the performance of all participants is compared. See the QA department for the results of recent PT studies.

13.0 Pollution Control

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i. e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention."

14.0 Waste Management

Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are managed, stored, and disposed of in accordance with all federal and state laws and regulations. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to the QA Manual and SOP Waste Disposal / NV10-83.

15.0 References / Cross-References

15.1 EPA Method 7470A, SW-846 Revision 1, September 1994.
15.2 EPA Method 7000B, SW-846 Revision 2, February 2007.
15.3 TestAmerica Nashville's Quality Assurance Manual.
15.4 TestAmerica Nashville's Control Limits Manual.
15.5 Corporate Environmental Health and Safety Manual (CW-E-M-001)
15.6 SOPs: Waste Disposal / NV10-83, Training Procedures for Environmental Technical Staff / NV08-199, Determination of Method Detection Limits / NV08-202

16.0 Method Modifications

None.

17.0 Attachments

None.

18.0 Revision History

- Revision 10, 15 May 2009
  - Integration of STL and TestAmerica formats.
  - Correction of acid addition volumes to samples based on 50-mL final volume.
- Revision 11, 25 September 2009

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- Incorporation of OH VAP requirements.
- Revision of preparation of LCS, MS/MSD, ICV, CCV, and standards due to change of pipettors.

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Title: INDUCTIVELY COUPLED PLASMA-ATOMIC EMISSION SPECTROMETRY SW-846 METHOD 6010B/C

Approvals (Signature/Date):

Rodney Street 10.29.09
Rodney Street Date
Department Supervisor

Michael H. Dunn 10.29.09
Michael H. Dunn Date
Technical Director

Eric S. Smith 10/29/09
Eric S. Smith Date
Quality Assurance Director

Jared Knierim 10-29-09
Jared Knierim Date
Health & Safety Manager / Coordinator

Johnny A. Mitchell 10.29.09
Johnny A. Mitchell Date
Laboratory Director

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1.0 Scope and Application

1.1 Analyte, Matrices: The method is applicable to the elements listed below. All matrices, excluding filtered groundwater samples but including ground water, aqueous samples, TCLP and SPLP extracts, industrial and organic wastes, soils, sludges, sediments, and other solid wastes, require digestion prior to analysis. Groundwater samples that have been pre-filtered and acidified do not need acid digestion. Refer to SOPs Method 3010 / NV06-18, 3015 / NV06-19, Method 3050 / NV06-93, 3051 / NV06-94, and 3005 / NV06-103 for the appropriate digestion procedures.

Table 1: Recommended Wavelengths and Typical Reporting Limits

Element[a]	Wavelength[a] (nm)	Typical RL (µg/L)	Typical RL (mg/kg)	CAS No.
Aluminum	308.215	100	10	7429-90-5
Antimony	206.833	10	10	7440-36-0
Arsenic	189.0	10	1.0	7440-38-2
Barium	493.4	10	2.0	7440-39-3
Beryllium	313.042	4	1.0	7440-41-7
Boron	249.678x2	50	10	7440-42-8
Cadmium	226.502	1	1.0	7440-43-9
Calcium	317.933 ICP1,2,4 323.7 ICP4	1000	10	7440-70-2
Chromium	267.716	5	1.0	7440-47-3
Cobalt	228.616	20	1.0	7440-48-4
Copper	324.754	10	2.0	7440-50-8
Iron	317.933 ICP1,2,4 323.7 ICP4	50	10	7439-89-6
Lead	220.353	5	1.0	7439-92-1
Lithium	670.784	50	10	7439-93-2
Magnesium	279.079	1000	10	7439-95-4
Manganese	257.610	15	1.0	7439-96-5
Molybdenum	202.030	50	3.0	7439-98-7
Nickel	231.604x2	10	1.0	7440-02-0
Potassium	766.491	1000	100	7440-09-7
Selenium	196.026	10	2.0	7782-49-2
Silver	328.068	5	1.0	7440-22-4
Sodium	588.995 ICP1, ICP4. 330.2 ICP2, ICP4.	1000	200	7440-23-5
Strontium	421.5	50	10	7440-24-6
Thallium	190.864	10	2.0	7440-28-0
Tin	189.980x2	50	10	7440-31-5
Titanium	334.941	50	10	7440-32-6
Vanadium	292.402	20	10	7440-62-2
Zinc	213.856x2 ICP1, ICP4. 206.2 ICP2	50	10	7440-66-6
Yttrium	371.03	IS	IS	7440-65-5
Scandium	361.384	IS	IS	7440-20-2
Indium	230.6	IS	IS	7440-74-6

[a]The wavelengths listed (where x2 indicates second order) are recommended because of their sensitivity and overall acceptance.

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1.2 Reporting Limits: Detection limits and linear concentration ranges of the elements can vary with the wavelength, spectrometer, matrix and operating conditions. Table 1 lists the analytical wavelengths and typical reporting limits in clean matrices.

1.3 Use of this method is restricted to analysts who are knowledgeable in the correction of spectral, chemical, and physical interferences described in this method.

1.4 The intent of the words "must," "shall," "should," or "will" renders the statement a mandatory requirement of this SOP.

1.5 If for any reason a part of this SOP cannot be followed, seek the guidance of the Department Supervisor or the Laboratory Technical Director. All abnormalities must be noted on the data or the benchsheet, in the Non-conformance database, and in LIMS. See the corrective action policies and Section 9 of this document.

2.0 Summary of Method

2.1 Prior to analysis, samples are solubilized or digested using appropriate Sample Preparation Methods. **When analyzing groundwater samples for dissolved constituents, acid digestion is not necessary if the samples are filtered and acid preserved prior to analysis.**

2.2 This method describes multi-elemental determinations by ICP-AES using simultaneous and sequential systems. Samples are nebulized and the resulting aerosol is transported to the plasma torch. Element-specific emission spectra are produced by a radio-frequency inductively coupled plasma. The spectra are dispersed by a grating spectrometer, and the intensities of the emission lines are monitored by photosensitive devices. Background correction is required for trace element determination. Background is measured adjacent to analyte lines on samples during analysis. The position selected for the background-intensity measurement, on either or both sides of the analytical line, is determined by the complexity of the spectrum adjacent to the analyte line. Background correction is not required in cases of line broadening where a background correction measurement would actually degrade the analytical result.

3.0 Definitions

3.1 Field Reagent Blank: An aliquot of reagent water that is placed in a sample container in the laboratory and treated as a sample in all respects, including shipment to the sampling site, exposure to the sampling site conditions, storage, preservation, and all analytical procedures. The purpose is to determine if method analytes or other interferences are present in the field environment.

3.2 Instrument Detection Limits (IDLs) are useful means to evaluate the instrument noise level and response changes over time for each analyte from a series of reagent blank analyses to obtain a calculated concentration. They are not to be confused with the lower limit of quantitation, nor should they be used in establishing this limit.

3.3 Internal Standard: Pure analyte added to a sample, in known amount(s) and used to measure the relative responses of other method analytes that are components of the same sample or solution. The internal standard must be an analyte that is not a sample component.

3.4 Linear Range (LR): The concentration range over which the instrument response to an analyte is linear.

3.5 Spectral Interference Check (SIC) Solution: Used to prepare ICSA and ICSAB. A solution of selected method analytes of higher concentrations which is used to evaluate the procedural routine for correcting known inter-element spectral interferences with respect to a defined set of method criteria. See Thermo Jarrell Ash method 136972-00 revision 0 (7/28/95) for procedure.

3.6 Inter-element Correction (IEC): Single element solutions are used to determine the appropriate location for background correction and to establish the inter-element correction routine.

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3.7 Toxicity Characteristics Leaching Procedure (TCLP): An extraction process which attempts to simulate the leaching of samples into the ground/soil at a municipal landfill.

3.8 TCLP Blank Matrix: An aliquot of TCLP fluid that is treated exactly as a sample including exposure to all glassware, equipment, solvents, reagents, and internal standards that are used with other samples. This blank is used to determine if method analytes or other interferences are present in the TCLP extraction fluid used in the preparation of TCLP samples.

3.9 Synthetic Precipitation Leaching Procedure (SPLP): An extraction process which attempts to simulate the leaching of samples in to the soil from a rain event.

3.10 SPLP Blank Matrix: An aliquot of SPLP fluid that is treated exactly as a sample including exposure to all glassware, equipment, solvents, reagents, and internal standards that are used with other samples. This blank is used to determine if method analytes or other interferences are present in the SPLP extraction fluid used in the preparation of SPLP samples.

3.11 See TestAmerica Nashville's QA Manual Appendix 5 for other laboratory definitions.

4.0 Interferences

4.1 Spectral interferences are caused by background emission from continuous or recombination phenomena, stray light from the line emission of high concentration elements, overlap of a spectral line from another element, or unresolved overlap of molecular band spectra.

4.1.1 Background emission and stray light can usually be compensated for by subtracting the background emission determined by measurements adjacent to the analyte wavelength peak. The locations selected for the measurement of background intensity are determined by the complexity of the spectrum adjacent to the wavelength peak. The locations used for routine measurement must be free of off-line spectral interference (inter-element or molecular) or adequately corrected to reflect the same change in background intensity as occurs at the wavelength peak.

4.1.2 Spectral overlaps may be avoided by using an alternate wavelength or can be compensated by equations that correct for inter-element contributions. Instruments that use equations for inter-element correction require the interfering elements be analyzed at the same time as the element of interest. When operative and uncorrected, interferences produce false positive determinations and are reported as analyte concentrations. Analysts may apply inter-element correction equations determined on their instruments with tested concentration ranges to compensate for the effects of interfering elements. Some potential spectral interferences observed for the recommended wavelengths are given in Table 2. The interferences listed are only those that occur between method analyses. Only interferences of a direct overlap nature are listed.

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Table 2: Potential Interferences by Analyte

Analyte	Wavelength (nm)	Interferant									
		Al	Ca	Cr	Cu	Fe	Mg	Mn	Ni	Ti	V
Aluminum	308.215	--	--	--	--	--	--	XX	--	--	XX
Antimony	206.833	XX	--	XX	--	XX	--	--	--	XX	XX
Arsenic	189.0	XX	--	XX	--	--	--	--	--	XX	
Barium	493.4	--	--	--	--	--	--	--	--	--	--
Beryllium	313.042	--	--	--	--	--	--	--	--	XX	XX
Cadmium	226.502	--	--	--	--	XX	--	--	XX	--	--
Calcium	317.933	--	--	XX	--	XX	XX	XX	--	XX	XX
Chromium	267.716	--	--	--	--	XX	--	XX	--	--	XX
Cobalt	228.616	--	--	XX	--	XX	--	--	XX	XX	--
Copper	324.754	--	--	--	--	XX	--	--	--	XX	XX

Analyte	Wavelength (ηm)	Interferant									
		Al	Ca	Cr	Cu	Fe	Mg	Mn	Ni	Ti	V
Iron	271.4	--	--	--	--	--	--	XX	--	--	--
Lead	220.353	XX	--	--	--	--	--	--	--	--	--
Magnesium	279.079	--	XX	XX	--	XX	--	XX	--	XX	XX
Manganese	257.610	XX	--	XX	--	XX	XX	--	--	--	--
Molybdenum	202.030	XX	--	--	--	XX	--	--	--	--	--
Nickel	231.604	--	--	--	--	--	--	--	--	--	--
Selenium	196.026	XX	--	--	--	XX	--	--	--	--	--
Sodium	588.995 / 330.2	--	--	--	--	--	--	--	--	XX	--
Thallium	190.864	XX	--	--	--	--	--	--	--	--	--
Vanadium	292.402	--	--	XX	--	XX	--	--	--	XX	--
Zinc	213.856 / 206.2	--	--	--	XX	--	--	--	XX	--	--
Dashes indicate that no interference was observed even when interferants were introduced.											
XX - Interference may be possible.											

4.1.3 When using inter-element correction equations, the interference may be expressed as analyte concentration equivalents (i. e., false analyte concentrations) arising from the linear range of the interference element. Instruments may exhibit somewhat different levels of interference than those shown in Table 2. The interference effects **must** be evaluated for each individual instrument since the intensities vary. This evaluation is filed in the method of the instrument's computer.

4.1.4 Inter-element correction accuracy must be verified daily by analyzing spectral interference check solutions. Inter-element correction factors must be verified every **six** months or when ICSA or ICSAB fail(s) criteria.

4.2 Physical interferences are effects associated with the sample nebulization and transport processes. Physical interferences are reduced by diluting the sample and by using an internal standard.

4.3 Chemical interferences include molecular compound formation, ionization effects, and solute vaporization effects. Normally, these effects are not significant with the ICP technique, but if observed, can be minimized by careful selection of operating conditions (incident power, observation position, and so forth), by matrix matching. Chemical interferences are highly dependent on matrix type and the specific analyte element.

4.4 Memory interferences result when analyses in a previous sample contribute to the signals measured in a new sample. Memory effects can result from sample deposition on the uptake tubing to the nebulizer and from the build up of sample material in the plasma torch and spray chamber. The site where these effects occur is dependent on the element and can be minimized by flushing the system with a rinse blank between samples. This method requires **a rinse period of at least 45 seconds between samples and standards**.

5.0 Safety

Employees must abide by the policies and procedures in the Corporate Safety Manual and this document.

This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

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5.1 Specific Safety Concerns or Requirements:

- The acidification of samples containing reactive materials may result in the release of toxic gases, such as cyanides or sulfides. Acidification of samples should be done in a fume hood.
- The inductively coupled plasma should only be viewed with proper eye protection from the ultraviolet emissions.
- The ICP uses high voltage.

5.2 Primary Materials Used: The following is a list of the materials used in this method, which have a serious or significant hazard rating. **Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Hydro-chloric Acid	Corrosive Poison	5 ppm-Ceiling	Inhalation of vapors causes coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Causes redness, pain, and severe skin burns. Vapors are irritating and cause damage to the eyes. Contact causes severe burns and permanent eye damage.
Nitric Acid	Corrosive Oxidizer Poison	2 ppm-TWA 4 ppm-STEL	Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison. Inhalation of vapors causes breathing difficulties and leads to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract. Causes redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and cause damage to the eyes. Contact causes severe burns and permanent eye damage.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

6.0 Equipment and Supplies

6.1 Instrumentation

- Inductively coupled plasma emission spectrometer, Thermo Jarrell-Ash 61E Trace, Thermo Scientific ICAP 6000 Series, or equivalent.
  - Computer-controlled emission spectrometer with background- correction capability. The spectrometer must be capable of meeting and complying with the requirements described and referenced in Section 4.0.
  - Radio-frequency generator compliant with FCC regulations.
  - Argon gas supply - High purity grade (99.99%).
  - A peristaltic pump is required to deliver both internal standard and sample solutions to the nebulizer.
  - Mass flow controller to regulate the argon flow rate, especially the aerosol transport gas.
  - See Table 3 for instrument operating conditions selected as being optimal to provide the lowest reliable instrument detection limits and method detection limits.

Table 3: Inductively Coupled Plasma Instrument Operating Conditions

Incident rf power	1100-1200 watts
Reflected rf power	< 5 watts

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Viewing height above work coil	15 mm
Injector tube orifice i.d.	1 mm
Argon supply	liquid argon
Argon pressure	40 psi
Coolant argon flow rate	15-19 L/min
Aerosol carrier argon flow rate	620-1000 mL/min
Auxiliary (plasma) argon flow rate	300 mL/min
Sample uptake rate controlled to	1-1.8 mL/min
Measurement time	10 seconds

- Specific wavelengths are listed in Table 1.
- Profiling is performed by introducing an Arsenic solution in the plasma. The selected wavelength is scanned and centered. Only one spectral line (As) is profiled; all other lines are preset relative to the profiled line. The interactive automatic profiling procedure is used to maintain the profile as close to center as possible. In this procedure, the spectrum shift is used to scan the profile line. The operator then instructs the computer to calculate the new profile Vernier setting, which centers the line. The operator then adjusts the profile Vernier to the new setting as the first step in daily operation.
  - For optimization in the axial mode, follow the instrument manufacturer's instructions.
  - The instrument operating condition finally selected as being optimum should provide the lowest reliable instrument detection limits and method detection limits.
  - If either the instrument operating conditions, such as incident power or nebulizer gas flow rate are changed, or a new torch injector tube with a different orifice internal diameter is installed, the plasma should be re-optimized.
  - A peristaltic pump must be used to introduce all solutions to the nebulizer. To allow equilibrium to be reached in the plasma, aspirate all solutions for 45 seconds after reaching the plasma before beginning integration of the background corrected signal to accumulate data.
- Analytical balance, with capability to measure to 1 mg.

6.2 Supplies

For determination of trace levels of elements, contamination and loss are of prime consideration. Potential contamination sources include improperly cleaned laboratory apparatus and general contamination within the laboratory environment from dust, etc. Sample containers can introduce positive and negative errors in the determination of trace elements by (1) contributing contaminants through surface desorption or leaching, (2) depleting element concentrations through adsorption processes. All reusable labware (glass, quartz, polyethylene, PTFE, FEP, etc.) must be sufficiently clean for the task objectives.

- Volumetric flasks, 25 mL, 100 mL, 200 mL, Class A.
- Automatic pipettors, 50 µL, 100 µL, 250 µL, 500 µL, 1000 µL, with disposable plastic tips.
- Disposable serological pipettes, 1 mL, 5 mL, 10 mL.
- Graduated Cylinders, 50 mL, 250 mL, 500 mL, Class A.
- Beakers, 150 mL, with ribbed watch glass.
- Centrifuge tubes, plastic, 50 mL, certified, graduated, with screw caps.
- Watch glass, plastic, ribbed (for use with the centrifuge tubes).
- Plastic centrifuge tube racks.
- Narrow-mouth storage bottles, FEP (fluorinated ethylene propylene) with screw closure, 125 L to 1-L capacities.
- One-piece stem FEP wash bottle with screw closure, 125 L capacity.

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- pH test strips.
- Teflon™ boiling chips for solid matrix blank (Chemware P/N D1069103, or equivalent).

7.0 Reagents and Standards

7.1 Reagent water, analyte-free, ASTM Type II or equivalent, used for diluent water.

7.2 Reagents may contain elemental impurities, which might affect analytical data. Only high-purity reagents that conform to the American Chemical Society specifications are used. If the purity of a reagent is in question, analyze for contamination. All acids used for this method **must be** of ultra high-purity grade or equivalent.

7.3 Hydrochloric acid, concentrated (sp.gr. 1.19), HCl. To prepare a 5% solution, add 10 mL concentrated HCl to 190 mL reagent water.

7.4 Nitric acid, concentrated (sp.gr. 1.41), HNO_3. To prepare a 5% solution, add 10 mL concentrated HNO_3 to 190 mL reagent water.

7.5 Standard Stock Solutions (HIGH and LOW calibration solutions are prepared from these; see Table 8): Stock standards are purchased as certified commercial solutions at 100, 500 or 1000 µg/mL (recommended). Stock solutions are stored in FEP bottles. Replace stock standards yearly when succeeding dilutions for preparation of calibration standards cannot be verified. Store standards containing silver in the dark.

7.6 Spectral Interference Check solutions are prepared in the same acid mixture as the calibration standards and stored in FEP bottles. See Table 6 for SIC Preparation (ICSA and ICSAB).

7.7 Internal Standards: Yttrium (ICP1), Scandium (for ICP2), Yttrium and Indium for ICP4. Purchase Ultra Scientific, or equivalent, commercial standards at 1000 µg/mL in 2% HNO_3: IAA-249-5 for Indium; IAA-239-5 for Yttrium; IAA-221-5 for Scandium. Dilute 5 mL to 1 liter with 5% HNO_3 for ICP1 and ICP2. For ICP4, dilute 5 mL Yttrium and 30 mL Indium to 1 liter with 5% HNO_3.

7.8 Inter-element Correction (IEC) Single-element Standards: Purchase the following single-element standards at 1000 µg/mL (Ca, Fe, Mg, Na at 10,000 µg/mL) from Ultra Scientific, or equivalent:

Element	Catalog #	Element	Catalog #	Element	Catalog #
Aluminum	ICP-013	Iron	ICP-026	Silver	ICP-047
Antimony	ICP-051	Lead	ICP-082	Sodium	ICP-111
Arsenic	ICP-033	Lithium	ICP-003	Strontium	ICP-038
Barium	ICP-056	Magnesium	ICP-112	Thallium	ICP-081
Beryllium	ICP-004	Manganese	ICP-025	Thorium	ICP-090
Boron	ICP-005	Molybdenum	ICP-042	Tin	ICP-050
Cadmium	ICP-048	Nickel	ICP-028	Titanium	ICP-022
Calcium	ICP-120	Phosphorus	ICP-015	Uranium	ICP-092
Chromium	ICP-024	Potassium	ICP-019	Vanadium	ICP-023
Cobalt	ICP-027	Selenium	ICP-034	Zinc	ICP-030
Copper	ICP-029	Silicon	ICP-014	Zirconium	ICP-040

7.9 See SOP Reagent and Standard Purchase, Preparation, Control, Documentation / NV08-214 for shelf-life and storage requirements for reagents and standards.

8.0 Sample Collection, Preservation, Shipment and Storage

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Matrix	Sample Container	Min. Sample Size	Preservation	Holding Time	Reference
Water, TCLP Extract	HDPE or Glass	125 mL	HNO_3 to pH ≤ 2[1]	6 months	SW-846 Chapter 2
Soil	HDPE or Glass	50 grams	No requirement	6 months	SW-846 Chapter 2

[1]If water samples are preserved in the lab, they should be held for at least 24 hours before analysis; record acidification start/stop time and pH. Temperature preservation is not required.

9.0 Quality Control

Refer to the quality control section of TestAmerica-Nashville's QA Manual for specific quality control (QC) policies. The laboratory maintains a formal quality assurance program and records to document the quality of the data generated.

9.1 Sample QC

The following quality control samples must be prepared with each batch of no more than 20 samples (or 10 samples if they are from OK or WY):			
Quality Controls	**Frequency**	**Acceptance Criteria**	**Corrective Action**
Method Blank	1 each batch	< ½ RL or MDL, whichever is greater.[2]	Re-analyze. If contamination persists, correct problem then re-prep and analyze method blank and all samples processed with the contaminated blank.
Laboratory Control Sample (LCS)[1], second source	1 each batch	80-120%[3] recovery	Correct problem then re-prep and analyze the LCS and all affected targets in the affected analytical batch. If high and ND, OK to report. For 6010C, LCS may be re-analyzed once.
Matrix Spike	1 each batch	75-125%[3] recovery	Perform post-digestion spike.
Matrix Spike Duplicate	1 each batch	75-125%[3] recovery <20[2]% RPD	Perform post-digestion spike.
Post digestion spike addition	When MS/MSD fail.	Recovery within 20% of the expected results	Perform dilution test.
Dilution test	If MS/MSD fail.	1:4 fold dilution (5X) must agree within 10% of the original determination.	Qualify results.

[1]AZ, TX, WV require an LCS duplicate in each batch.
[2]Report all targets above the MDL.
[3]If historical limits are calculated, they cannot exceed these limits.

- **Method Blank:** The laboratory prepares and analyzes one blank with each batch of up to 20 samples of the same matrix. Blank values that exceed the MDL indicate laboratory or reagent contamination. The method blank must not contain any analyte of interest at a level greater than ½ the reporting limit (RL) or MDL, whichever is greater, for that analyte. If any analyte is detected at a level exceeding the criterion, re-analyze the blank. If the contamination is persistent, all samples in the batch must be re-digested.
- A **Laboratory Control Sample (LCS)** must be analyzed with every batch. If the recovery of any analyte falls outside the required control limits of **80-120%**, that analyte is judged out of control, and the source of the problem is identified and resolved before continuing analyses. Re-prep and re-analyze the batch. However, if it fails high and the result is not detected, it is acceptable to report the result. For 6010C, the LCS may be re-analyzed once. If it fails

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again, corrective action is required. See Table 4 for LCS preparation using 50 mL reagent water for water batches and 0.5 gram Teflon™ chips for soil batches.

- **Matrix Spike / Matrix Spike Duplicate:** Sample homogeneity and the chemical nature of the sample matrix can affect analyte recovery and the quality of the data. Taking separate, identical aliquots from the sample for replicate and spiked analyses can in some cases assess the effect.
  - Analyze a matrix spike and matrix spike duplicate at a frequency of one per matrix batch up to 20 samples. In each case the MS aliquot must be a duplicate of the aliquot used for sample analysis and for total recoverable determinations added prior to sample preparation. See SOP Sample Homogenization, Subsampling, and Compositing / NV08-229. The added analyte concentration and standard source must be the same as that used in the LCS (Table 4).
  - Calculate the percent recovery for each analyte (see Section 11), corrected for background concentrations measured in the unspiked sample, and compare these values to the designated MS recovery range of 75-125%. A control limit of ± 20% RPD for each matrix is used for sample values greater than ten times the RL.
  - If the recovery of any analyte falls outside the designated MS recovery range, and the LCS for that analyte is shown to be in control, the recovery problem encountered with the spiked sample is judged to be matrix-related, not system-related. The data user is informed that the result for that analyte in the unspiked sample is suspect due to either the heterogeneous nature of the sample or matrix effects by flagging the MS recovery on the final report.
  - For each batch, check for matrix effects as follows:
    - If MS/MSD is outside the QC limits, the same sample from which the MS/MSD aliquots were prepared is also spiked with a **post-digestion spike** (i. e., nominally add 250 µL of the LCS/MS/MSD solution to 25 mL digested aliquot). Otherwise, another sample from the same preparation is used as an alternative. An analyte spike is added to a portion of a prepared sample, or its dilution, and is recovered to within 80-120% of the known value. The spike addition produces a minimum level of 10 times and a maximum of 100 times the lower limit of quantitation. If this spike fails, then the dilution test is run on this sample. If both the MS/MSD and the post-digestion spike fail, then matrix effects are confirmed.
    - **Dilution test**: If the analyte concentration is sufficiently high (minimally, a factor of 10 above the instrumental detection limit after dilution), an analysis of a 1:4 dilution (5X) should agree within 10% of the original determination. If not, a chemical or physical interference effect is suspected.

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9.2 Instrument QC

Quality Controls	Frequency	Acceptance Criteria	Corrective Action
Inter-element Correction, single element standards	6 months	± RL	Alter wavelength or background correction.
Spectral Interference Check Solutions, A and AB	Beginning and end of each day or every 8 hours	Target ± 2 times RL or ± 20% true.	Terminate analysis; correct problem; re-analyze ICS; re-analyze all affected samples.
Instrument Detection Limits (IDL)	Quarterly	±3 standard deviations of the average response.	Re-run IDL. If > MDL, adjust MDL to equal IDL.
Independent Calibration Verification Sample (ICV), second source	Immediately after calibration	90-110 % recovery	Correct problem then repeat initial calibration.

Quality Controls	Frequency	Acceptance Criteria	Corrective Action
Independent Calibration Blank (ICB)	Immediately after ICV	No target analytes above MDL or ½ RL, whichever is greater.	Correct problem, re-calibrate.
Linear Range Standard	Daily	90-110%	Repeat calibration (ICP1 and ICP2).
Continuing Calibration Verification Sample (CCV)	Every 10 samples and at the end of the run	90-110% true	Re-analyze once. If fails again, then repeat calibration and re-analyze all samples since last successful CCV.
Undigested Low Level Continuing Calibration Verification (LLCCV)	Beginning and end of each batch.	70-130% true	Re-calibrate.
Continuing Calibration Blank	Following the CCV	< IDL or MDL	Correct problem then analyze calibration blank and previous 10 samples.
MDL Verification (digested)	Yearly	Detected	Re-evaluate MDL standard used and MDL; see Technical Director
Digested Lower Limit of Quantitation Check (LLQC) or Report Limit Verification (RLV)	Once daily	70-130% true	Re-calibrate.
Internal Standards	All samples, standards, QC	60-140% true	Dilute and re-run. For blank and LCS, correct problem and re-run batch.

- **Inter-element Correction (IEC):** When correction is appropriate, the concentration of all targets must be within ± the RL, i. e., RL = 0.01, acceptance is -0.01 to +0.01. Once established, the entire routine must be initially and periodically verified every six months, or whenever there is a change in instrument operating conditions or when ICSA or ICSAB fail criteria.

  Single element standard concentrations that must be analyzed every six months are shown as follows:

Element	Required Final Concentration (mg/L)	Stock Standard Solution (µg/mL)	Volume of Stock Standard Solution (mL)	Final Volume (mL)
Aluminum	1000	1000	Use as received	
Antimony	50	1000	2.5	50
Arsenic	50	1000	2.5	50
Barium	50	1000	2.5	50
Beryllium	20	1000	1.0	50
Boron	100	1000	5.0	50
Cadmium	25	1000	1.25	50
Calcium	2000	10,000	10.0	50
Chromium	50	1000	2.5	50
Cobalt	50	1000	2.5	50
Copper	50	1000	2.5	50
Iron	2000	10,000	10.0	50
Lead	200	1000	10.0	50

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Element	Required Final Concentration (mg/L)	Stock Standard Solution (µg/mL)	Volume of Stock Standard Solution (mL)	Final Volume (mL)
Lithium	25	1000	1.25	50
Magnesium	1500	10,000	7.5	50
Manganese	20	1000	1.0	50
Molybdenum	20	1000	1.0	50
Nickel	50	1000	2.5	50
Phosphorus	50	1000	2.5	50
Potassium	500	1000	25.0	50
Selenium	50	1000	2.5	50
Silicon	200	1000	10.0	50
Silver	2	1000	0.1	50
Sodium	10000	10,000	Use as received	
Strontium	20	1000	1.0	50
Thallium	50	1000	2.5	50
Thorium	50	1000	2.5	50
Tin	50	1000	2.5	50
Titanium	25	1000	1.25	50
Uranium	50	1000	2.5	50
Vanadium	50	1000	2.5	50
Zinc	50	1000	2.5	50
Zirconium	50	1000	2.5	50

If interferences are observed, they must be mitigated by the use of interference correction equations or by changing to a different wavelength. The objective is to reduce interferences.

Required ICSA and ICSB solutions are described in Table 6 for daily verification. Initial and periodic verification data of the routine are filed in the maintenance of the instrument's computer.

- **Spectral interference check solution (ICSA and ICSAB):** The laboratory must periodically verify the inter-element correction routine by analyzing SIC solutions. The spectral interference check solution is run at the beginning and end of the day's sequence or every 8 hours, whichever is more frequent. If the SIC does not meet criteria, then the SICs are re-analyzed. These solutions are used to periodically verify a partial list of the on-line (and possible off-line) inter-element spectral correction factors for the recommended wavelengths given in Table 1.

  NOTE: The SIC solution must be analyzed more than once to confirm a change has occurred with adequate rinse time between solutions and before subsequent analysis of the calibration blank.

  - Ensure that the analytical results of ICS Solution A (ICSA) fall within the control limit of ± 2 times the RL of the analyte's true value or ± 20% of the analyte's true value, whichever is greater (the true value is zero unless otherwise stated) in the ICSA. For example, if the analysis result(s) for Arsenic (RL = 10 µg/L, ICSA true value = 0 µg/L) in the ICSA analysis during the run is 19 µg/L, then the analytical result for Arsenic falls within the ± 2 times the RL window for Arsenic in the ICSA. If the analytical results of the ICSA do not fall within the control limits, terminate the analysis, correct the problem, re-calibrate the

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instrument, and re-analyze the analytical samples analyzed since the last compliant ICSA was performed.

- Ensure that the results for the ICS Solution AB (ICSAB) during the analytical runs fall within the control limit of ± 2 times the RL of the true value or ± 20% of the true value, whichever is greater, for the analytes included in the ICSAB. If the analytical results of the ICSAB do not fall within the control limits, terminate the analysis, correct the problem, re-calibrate the instrument, and re-analyze the analytical samples analyzed since the last compliant ICSAB was performed.

- **Instrument Detection Limits (IDL):** IDLs in µg/L are estimated by calculating the average of the standard deviations of three runs on three non-consecutive days from the analysis of a reagent blank solution with seven consecutive measurements per day. Perform each measurement as though it were a separate analytical sample (i. e., follow each measurement by a rinse and/or any other procedure normally performed between the analysis of separate samples). Determine IDLs at least every three months and keep with the instrument logbook. Compare the calculated IDLs to the MDLs. MDLs are equal to or greater than the IDL.
- **Independent Calibration Verification (ICV and ICB):** The laboratory analyzes the ICV and a calibration blank (ICB) immediately following daily calibration. See Table 7 for ICV preparation using a second-source standard. Analysis of the ICV must verify that the instrument is **within ± 10%** of known concentration. The ICB must not contain target analytes above the MDL or ½ RL, whichever is greater.
- **Linear Range Standard (LRS):** For single-point calibration, run at the beginning of the analytical sequence. Run the highest standard level, to show linearity to that concentration. All samples exceeding **90%** this concentration are diluted. The LRS concentrations are Al, Fe, Ca and Mg at 200 µg/mL, Na, K, at 100 µg/mL, Ba at 50 µg/mL, Ag at 5 µg/mL, and all other elements at 10 µg/mL. The standard must be within 10% of the true values to continue.
- **Undigested Low-Level Continuing Calibration Verification (LLCCV):** Analyze an undigested LLCCV at the beginning and end of each batch. Prepare it from the primary calibration standard at the RL. If not within **70-130%** of the true value, re-calibrate.
- **Continuing Calibration Verification (CCV and CCB):** Analyze after every 10th sample and at the end of the analytical sequence.
  - See Table 5 for CCV preparation using the primary source standard at the mid-point of the calibration curve. Analysis of the **CCV** solution must be within **± 10% of true value**. If the calibration cannot be verified within the specified limits, reanalyze the CCV and the CCB. If the second analysis of the CCV and the CCB confirm calibration to be outside the limits, sample analysis must be discontinued, the cause determined, corrected and/or the instrument recalibrated. All samples following the last acceptable CCV must be reanalyzed. All samples **must be bracketed** by acceptable CCVs and CCBs.
  - The CCB (prepared by adding 25 mL concentrated HNO_3 to 500 mL reagent water) must not contain target analytes above the MDL or ½ the RL, whichever is greater. If the criterion is not met, terminate the analysis, correct the problem, and re-analyze the previous 10 samples.
- **MDL Verification:** A solution containing all target analytes at 2-3 times the MDL must be **digested** and analyzed after the completion of the MDL study and on an annual basis. Detection limits are verified when all analytes in the MDL check solution are detected.
- **Lower Limit of Quantitation Check Sample (LLQC):** The lower limit of quantitation check (LLQC) sample is analyzed once daily. This check sample and the low-level calibration verification standard are prepared at the same concentrations (Table 8) with the only difference being the LLQC sample is carried through the digestion procedure. Lower limits of quantitation are verified when all analytes in the LLQC sample are within ± 30% of their true value.

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- **Internal Standards:** Use the internal standard technique by adding one or more elements (not in the samples and verified not to cause an uncorrected inter-element spectral interference) at the same concentration (which is sufficient for optimum precision) to the prepared samples (blanks and standards) that are affected the same as the analytes by the sample matrix. Use the ratio of analyte signal to the internal standard signal for calibration and quantitation. Internal standards (Yttrium and Scandium (and Indium for ICP4)) are automatically added to all calibration standards, samples, and QC, by the instrument. Acceptance criteria are 60% to 140% true as compared to the calibration blank.

10.0 Procedure

10.1 Sample Preparation

Preliminary treatment of most matrices is necessary because of the complexity and variability of sample matrices. Groundwater samples which have been pre-filtered and acidified do not need acid digestion. Samples which are not digested must use an internal standard. See Section 1.1 for digestion SOPs.

Matrix	Sample Size
Water	50 mL of sample
TCLP Extract	10 mL of extract
Soil	0.5 gram of sample

10.2 Instrument Setup

1	Turn power ON to unit, choose appropriate item number. Allow the instrument to become thermally stable before beginning (usually requiring at least 30 minutes of operation prior to calibration).
2	Connect peristaltic tubing, turn ON fan, select "set-up" from menu and select "plasma", press F1 then F9.
3	Confirm flow to plasma.
4	Select operating method and profile instrument by pressing "profile" from the main menu.
5	To shut down ICP return to plasma control and press F7, after 60 seconds turn OFF fan and release pump tubing.

10.3 Calibration

See Section 11 for equations.	
1	See Table 8 for calibration standard preparation. For single-point calibration, the initial calibration curve is prepared daily with a minimum of a calibration blank and a single high standard (ICP1 and ICP2). The quantitation limit cannot be reported lower than the LLCCV. For multi-point calibration (ICP4), use first-order linear regression (**$r \geq 0.998$, $r^2 \geq 0.996$**). The lowest non-zero standard concentration is considered the lower limit of quantitation, i. e., RL. Higher order fits are not allowed.
2	The absolute value of the results of the calibration blank is less than the value of the MDL or ½ RL, whichever is greater, check sample for each analyte or less than the level of acceptable blank contamination specified in the approved quality assurance project plan.
3	After initial calibration, the calibration curve must be verified by use of an initial calibration

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	verification (ICV) standard. The calibration curve must be verified at the end of each analysis batch and after every 10 samples by use of a continuing calibration verification (CCV) standard and a continuing calibration blank (CCB).
4	The calibration curve must also be verified prior to the analysis of any samples and at the end of the batch by use of a low-level continuing calibration verification (LLCCV) standard.
5	Run LRS standards and evaluate by the criteria in Section 9.
6	Verify the inter-element correction factors at the beginning and end of the daily sequence or every 8 hours.

10.4 Sample Analysis

- Follow the analysis sequence in Table 9.
- Samples exceeding the 90% of the LRS are diluted and rerun.

11.0 Calculations / Data Reduction

11.1 Accuracy

$$\textbf{\% Recovery} = \frac{\text{Measured concentration}}{\text{Known concentration}} \times 100$$

11.2 Precision (RPD)

$$\textbf{RPD} = \frac{\text{Absolute value (orig. sample value - dup. sample value)} \times 100}{\text{(Orig. sample value + dup. sample value)/2}}$$

11.3 Response Factor

$$RF = \frac{A_s x C_{is}}{A_{is} x C_s}$$

A_s = Response of the analyte.
A_{is} = Response of the internal standard.
C_s = Concentration of the analyte.
C_{is} = Concentration of the internal standard

11.4 % Drift

$$\textbf{\% Drift} = \frac{\text{(Result - True Value)} \times 100}{\text{True Value}}$$

11.5 Linear Calibration Using a Least Squares Regression: This is most easily achieved by performing a linear regression of the instrument response versus the concentration of the standards. Make certain that the instrument response is treated as the dependent variable (y) and the concentration as the independent variable (x). This is a statistical requirement and is not simply a graphical convention.

The regression produces the slope and intercept terms for a linear equation in the form:

$$y = ax + b$$

y = instrument response (peak area)

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a = slope of the line
x = concentration of the calibration standard
b = the intercept

The regression calculation generates a correlation coefficient (r) that is a measure of the "goodness of fit" of the regression line to the data. A value of 1.00 indicates a perfect fit.

11.6 Coefficient of Determination Correlation Coefficient

$$r^2 = \frac{\left(\sum xy\right)^2}{\sum x^2 \sum y^2} \qquad r = \frac{\left(\sum xy\right)}{\sqrt{\sum x^2 \sum y^2}}$$

y = Response ratio
x = Concentration

11.7 Concentration: Sample data are reported in units of mg/L for aqueous samples, mg/kg for solid samples. LIMS calculates the concentration from the raw data provided by the analyst.

- For single-point calibration:

Concentration (mg/L or mg/kg) = $\frac{(\mu g/mL^* \text{ from instrument})(\text{digest volume, mL})(\text{Dilution factor})}{\text{Sample volume (mL) or Mass (g)}}$

µg/mL from instrument = y = mx + b

***average of two replicates**

- For multi-point calibration:

Concentration (mg/L or mg/kg) = $\frac{(\mu g/mL^* \text{ from instrument})(\text{digest volume, mL})(\text{Dilution factor})}{\text{Sample Volume (mL) or Mass (g)}}$

µg/mL from instrument = y = mx + b

***average of two replicates**

11.8 For dissolved aqueous analytes, report the data generated directly from the instrument with allowance for sample dilution. Do not report analyte concentrations below the MDL.

11.9 Account for any additional dilution of the prepared sample solution needed to complete the determination of analytes exceeding 90% or more of the LRS upper limit. Do not report data below the determined analyte MDL concentration or below an adjusted detection limit reflecting smaller sample aliquots used in processing or additional dilutions required to complete the analysis.

12.0 Method Performance

12.1 Method Detection Limit Study (MDL): The method detection limit (MDL) is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. The MDL is determined according to the laboratory's MDL procedure in SOP Determination of Method Detection Limits / NV08-202. MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix, and may not be achievable in all environmental matrices. The laboratory maintains MDL studies for analyses

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performed; these are verified at least annually unless method requirements require a greater frequency.

Quarterly, run 7 IDLs on three non-consecutive days; calculate the standard deviation per day. Compare three times the average of the standard deviations to the MDL. If 3xIDL > MDL, rerun the IDLs. If the finding persists, select the higher of the two as the "working MDL" for that quarter.

12.2 Demonstration of Capability: The laboratory demonstrates initial proficiency by generating data of acceptable accuracy and precision for target analyses in a clean matrix. The laboratory also repeats the operation whenever new staff is trained or significant changes in instrumentation are made and on an annual basis thereafter. See the training section of TestAmerica-Nashville's QA Manual and SOP Training / NV08-199 for information on how to accomplish this demonstration.

12.3 Training Requirements: Demonstration of Capability is performed initially when learning the method and annually thereafter. Four Laboratory Control Samples resulting in an average % recovery within the control limits and a precision less than the quality control maximum are required.

12.4 Proficiency Testing Studies: The laboratory participates in formal proficiency testing (PT) studies, where solutions of unknown concentrations are analyzed and the performance of all participants is compared. See the QA department for the results of recent PT studies.

12.5 Control Charts: Laboratory method performance can be shown with the use of control charts, available from LIMS or the QA department.

13.0 Pollution Control

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i. e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention."

14.0 Waste Management

Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are managed, stored, and disposed of in accordance with all federal and state laws and regulations. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to the QA Manual and SOP Waste Disposal / NV10-83.

15.0 References / Cross-References

15.1 Method 6010B, SW-846 Update III, Revision 2, December 1996, and Method 6010C, SW-846 Update IV, Revision 3, February 2007.

15.2 TestAmerica Nashville's Quality Assurance Manual.

15.3 TestAmerica Nashville's Control Limits Manual.

15.4 Corporate Environmental Health and Safety Manual (CW-E-M-001).

15.5 SOPs: Method 3005 / NV06-103; Method 3010 / NV3010, Method 3015 / NV06-19; Method 3050 / NV06-93; Method 3051 / NV06-94; Waste Disposal / NV10-83, Training Procedures for Environmental Technical Staff / NV08-199, Balance Calibration / NV08-213, Determination of Method Detection Limits / NV08-202, Sample Homogenization, Subsampling, and Compositing / NV08-229, Reagent and Standard Purchase / NV08-214.

16.0 Method Modifications

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Item	Modification
1	If 3030C digestion is specified, see the attachment in SOP 3005 / NV06-103 for that procedure.
2	For OK, NV, SC, and WY samples, reference 6010C. For all other states, reference 6010B.
3	Corporate Quality Memorandum CA-Q-QM-004, Technical Guidance on Checking for Spectral Interferences in Optical ICP Analysis, September 24, 2009.

17.0 Attachments

Table 4: LCS and MS/MSD Spiking Solution for ICP 1, ICP2, and ICP4
Environmental Express HP3540
For waters, use 0.5 mL; see water digestion methods.
For soils, use 2.0 mL; see soil digestion methods.

Analyte	Stock Std Concentration (µg/mL)	Final Concentration of Digestate (µg/mL)
Aluminum	200	2.0
Antimony	10	0.1
Arsenic	5	0.05
Barium	200	2.0
Beryllium	5	0.05
Bismuth	100	1.0
Boron	100	1.0
Cadmium	5	0.05
Calcium	500	5.0
Chromium	20	0.2
Cobalt	50	0.5
Copper	25	0.25
Iron	100	1.0
Lead	5	0.05
Lithium	100	1.0
Magnesium	500	5.0
Manganese	50	0.5
Molybdenum	50	0.5
Nickel	50	0.5
Potassium	500	5.0
Silver	5	0.5
Sodium	500	5.0
Thallium	5	0.05
Titanium	100	1.0
Vanadium	50	0.5
Zinc	50	0.5

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Table 5: Calibration Solutions

ICP1 and ICP2

ICAL solutions purchased from commercial source:
ICAL 1: **Ultra Scientific ICUS-1946** (100 µg/mL each element)
ICAL 2: **Ultra Scientific ICUS-1829** (500 µg/mL Al, Ba, 300 µg/mL Ba, 50 µg/mL Ag)

ICAL 3: **Ultra Scientific ICUS-1830** (100 µg/mL Ca, Mg, 1000 µg/mL Na, K)
ICAL 4: **CPI 4400-1000612** (1000 µg/mL Sn)
For the ICAL combined standard, add 10 mL ICAL 1, 2, 3 to 500 mL reagent water.
For the CCV combined standard. Add 5 mL ICAL 1, 2, 3 to 500 mL reagent water.

Analyte	ICAL 1 (µg/mL)	ICAL 2 (µg/mL)	ICAL 3 (µg/mL)	ICAL 4 (µg/mL)
Antimony	2.0			
Arsenic	2.0			
Beryllium	2.0			
Boron	2.0			
Cadmium	2.0			
Chromium	2.0			
Cobalt	2.0			
Copper	2.0			
Lead	2.0			
Lithium	2.0			
Manganese	2.0			
Molybdenum	2.0			
Nickel	2.0			
Selenium	2.0			
Strontium	2.0			
Thallium	2.0			
Titanium	2.0			
Vanadium	2.0			
Zinc	2.0			
Aluminum		10.0		
Barium		10.0		
Iron		6.0		
Silver		1.0		
Calcium			2.0	
Magnesium			2.0	
Potassium			2.0	
Sodium			2.0	
Tin				20

ICP4					
Analyte	**ICAL 1**	**ICAL 2**	**ICAL 3**	**ICAL 4**	**ICAL 5**
Al, Sb, Ar, Ba, Be, B, Cd, Co, Cr, Cu, Fe, Pb, Li, Mg, Mn, Mo, Nl, Ag, Tl, Sn, Ti, V, Zn	ICUS 2648: 2.0 µg/mL	Dilute 20 mL ICUS 2648/ 40 mL: 1.0 µg/mL	Dilute 10 mL ICUS 2648/ 40 mL: 0.5 µg/mL		
Ba	ICUS 2615: 10.0 µg/mL				
Al	ICUS 2615: 10.0 µg/mL	ICUS 2614: 500 µg/mL			
K	ICUS 2615: 10.0 µg/mL	ICUS 2614: 100 µg/mL			

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Analyte	ICAL 1	ICAL 2	ICAL 3	ICAL 4	ICAL 5
Mg,	ICUS 2615: 10.0 µg/mL	ICUS 2614: 500 µg/mL			
Ca	ICUS 2615: 10.0 µg/mL		5 mL 1000 µg Ca /mL stock/50 mL: 100 µg/mL	15 mL 1000 µg Ca /mL stock/50 mL: 300 µg/mL	ICUS 2614: 500 µg/mL
Fe	ICUS 2615: 10.0 µg/mL	2.5 mL 1000 µg Fe/mL stock/50 mL: 50 µg/mL	5 mL 1000 µg Fe /mL stock/ 50 mL: 100 µg/mL	ICUS 2614: 200 µg/mL	15 mL 1000 µg Fe /mL stock/ 50 mL: 300 µg/mL
Na	ICUS 2615: 10.0 µg/mL		5 mL 1000 µg Na /mL stock/ 50 mL: 100 µg/mL	15 mL 1000 µg Na /mL stock/ 50 mL: 300 µg/mL	ICUS 2614: 500 µg/mL

ICUS 2614 = LRS.

CCV and RLV Sources and Concentrations for ICP4

Analyte	CCV ICUS-2613 (µg/mL)	RLV ICUS-2647 (µg/mL)
Aluminum	10.0	0.1
Antimony	1.0	0.01
Arsenic	1.0	0.01
Barium	2.0	0.01
Beryllium	1.0	0.004
Boron	1.0	0.05
Cadmium	1.0	0.001
Calcium	10.0	1.0
Chromium	1.0	0.005
Cobalt	1.0	0.02
Copper	1.0	0.01
Iron	10.0	0.05
Lead	1.0	0.005
Lithium	1.0	0.05
Magnesium	10.0	1.0
Manganese	1.0	0.015
Molybdenum	1.0	0.05
Nickel	1.0	0.01
Potassium	10.0	1.0
Selenium	1.0	0.01
Silver	1.0	0.005
Sodium	10.0	1.0
Strontium	1.0	0.05
Thallium	1.0	0.01
Tin	1.0	0.05
Titanium	1.0	0.05

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Analyte	CCV ICUS-2613 (µg/mL)	RLV ICUS-2647 (µg/mL)
Vanadium	1.0	0.02
Zinc	1.0	0.05

Table 6: Spectral Interference Check Solutions

ICSA Standard

ICSA Stock, SPEX # INT-A1

ICSA Solution: Dilute 20 mL of ICSA Stock + 10 mL HNO_3 to 200 mL with reagent water.

Analyte	ICSA Stock Conc. (µg/mL)	ICSA Conc. (µg/mL)
Aluminum	5000	500
Calcium	5000	500
Magnesium	5000	500
Iron	2000	200

ICSAB Standard

ICSA Stock, SPEX # INT-A1
ICSB Stock, SPEX # INT-B1
ICSB2 Stock, SPEX # INT-B2

ICSAB Solution: ***Dilute 20 mL of ICSA Stock + 2 mL of ICSB Stock + 2 mL of ICSB2*** Stock + 10 mL HNO_3 to 200 mL with reagent water.

Analyte	ICSA Stock Conc. (µg/mL)	ICSB Stock Conc. (µg/mL)	ICSB2 Stock Conc. (µg/mL)	ICSAB Conc. (µg/mL)
Aluminum	5000		100	501
Calcium	5000		10	500.1
Magnesium	5000		10	500.1
Iron	2000		10	200.1
Barium		50		0.5
Beryllium		50		0.5
Cadmium		100		1.0
Chromium		50		0.5
Cobalt		50		0.5
Copper		50		0.5
Lead		100		1.0
Manganese		50		0.5
Nickel		100		1.0
Silver		100		1.0
Vanadium		50		0.5
Zinc		100		1.0
Antimony			100	1.0
Arsenic			100	1.0
Boron			100	1.0
Molybdenum			100	1.0

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Analyte	ICSA Stock Conc. (µg/mL)	ICSB Stock Conc. (µg/mL)	ICSB2 Stock Conc. (µg/mL)	ICSAB Conc. (µg/mL)
Selenium			100	1.0
Silica			10	0.1
Sodium			100	1.0
Thallium			100	1.0

Table 7: ICV Standard
Source: Inorganic Ventures STLTN-CAL-3 for All but Tin
Ultra Scientific 1AA-250, 1000 µg/mL for Tin
ICV Preparation: 0.05 mL Tin standard to 50 mL STLTN-CAL-3

Analyte	STLTN-CAL-3 (µg/mL)	Ultra 1AA-250 Sn (µg/mL)	ICV Conc. (µg/mL)
Aluminum	10		10
Antimony	1		1
Arsenic	1		1
Barium	2		2
Beryllium	1		1
Boron	1		1
Cadmium	1		1
Calcium	10		10
Chromium	1		1
Cobalt	1		1
Copper	1		1
Iron	10		10
Lead	1		1
Lithium	1		1
Magnesium	10		1
Manganese	1		1
Molybdenum	1		1
Nickel	1		1
Potassium	10		10
Selenium	1		1
Silver	1		1
Sodium	10		10
Strontium	1		1
Thallium	1		1
Tin		1000	1
Titanium	1		1
Vanadium	1		1
Zinc	1		1

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Table 8: HIGH and LOW Calibration Standards

All Stock Solutions are at 1000 µg/mL.
For High Standard: Dilute the indicated volumes + 12.5 mL HNO_3 to 250 mL reagent water
For Low Standard: Dilute the indicated volumes + 50 mL HNO_3 to 1000 mL reagent water.

	To Make High Standard		To Make Low Standard			
Analyte	**Stock Diluted to 250 mL Reagent Water (mL)**	**HIGH Std. Conc. (µg/mL)**	**Stock Diluted to 1L Reagent Water (mL)**	**Intermediate Conc. (µg/mL)**		**LOW Std Conc. (µg/mL)**
Arsenic	2.5	10	1.0	1.0		0.01
Barium	12.5	50	1.0	1.0		0.01
Cadmium	2.5	10	0.1	0.1		0.001
Chromium	2.5	10	0.5	0.5		0.005
Lead	2.5	10	0.5	0.5		0.005
Selenium	2.5	10	1.0	1.0		0.01
Silver	1.25	5	Not applicable	Not applicable		Not app.
Copper	2.5	10	1.0	1.0		0.01
Nickel	2.5	10	1.0	1.0	Dilute	0.01
Aluminum	12.5	50	10	10		0.1
Beryllium	2.5	10	0.4	0.4	Intermediate	0.004
Calcium	2.5	10	5.0	5.0		0.05
Cobalt	2.5	10	2.0	2.0	0.5 mL	0.02
Iron	7.5	30	5.0	5.0		0.05
Magnesium	2.5	10	5.0	5.0	to	0.05
Manganese	2.5	10	1.5	1.5		0.0015
Potassium	25.0	100	50	50	50 mL	0.5
Thallium	2.5	10	1.0	1.0		0.01
Vanadium	2.5	10	2.0	2.0	in	0.02
Zinc	2.5	10	5.0	5.0		0.05
Antimony	2.5	10	1.0	1.0	5% HNO_3	0.01
Molybdenum	2.5	10	0.5	0.5		0.005
Boron	2.5	10	5.0	5.0		0.05
Titanium	2.5	10	5.0	5.0		0.05
Sodium	25.0	100	50	50		0.5
Lithium	10	10	5.0	5.0		0.05
Strontium	2.5	10	5.0	5.0		0.05

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Additional Standards

Dilute the following volumes + *10 mL HNO_3* to 250 mL reagent water

Analyte	Stock Conc. (µg/mL)	Volume (mL) Diluted to 250 mL Reagent Water	Cal. Std. Conc. (µg/mL)
Aluminum	1000	50	200
Iron	1000	50	200
Calcium	1000	50	200
Magnesium	1000	50	200

Lead	1000	5 mL diluted to 50 mL reagent water + 2 mL HNO_3	100

ICP4: Use ICUS 2614, or equivalent.

Analyte	ICUS 2614 Conc. (µg/mL)
Al, Ca, Mg, Na	500
Fe	200
K	100

Table 9: Typical Analytical Sequence

Definitions

ICP1 and ICP2	
ICAL B	Initial Calibration Blank
ICP Calibration Standard	Initial Single Point Calibration (ICAL Combined Standard)
ICV	Independent (initial) Calibration Verification
ICB	Initial Calibration Blank
RLV	Report Limit Verification
LRS	High Range Calibration Verification
Linear Range Standard 1	Linear Range Standard
Linear Range Standard 2	Linear Range Standard
ICSA	Initial Spectral Interference Check
ICSAB	Initial Spectral Interference Check
MB	Method Blank
LCS	Laboratory Control Sample
10 samples	
CCV	Continuing Calibration Verification
CCB	Continuing Calibration Blank
Up to 10 samples	
MS	Matrix Spike
MSD	Matrix Spike Duplicate
RLV (as needed)	Report Limit Verification
CCV	Continuing Calibration Verification
CCB	Continuing Calibration Blank
ICSA	Spectral Interference Check (every 8 hours or less)
ICSAB	Spectral Interference Check (every 8 hours or less)

ICP4	
0.0 mg/L standard(or blank)	Calibration Standard
0.5 mg/L	Calibration Standard
1.0 mg/L	Calibration Standard
2.0 mg/L	Calibration Standard
10.0 mg/L	Calibration Standard
100 mg/L Cu, Fe	Calibration Standard
300 mg/L Na	Calibration Standard
50 mg/L Fe	Calibration Standard
ICV	Independent (initial) Calibration Verification
ICB	Initial Calibration Blank
RLV	Report Limit
ICSA	Initial Spectral Interference Check
ICSAB	Initial Spectral Interference Check
MB	Method Blank
LCS	Laboratory Control Sample

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Up to 10 samples	
CCV	Continuing Calibration Verification
CCB	Continuing Calibration Blank
Up to 10 samples	
MS	Matrix Spike
MSD	Matrix Spike Duplicate
RLV	Report Limit
CCV	Continuing Calibration Verification
CCB	Continuing Calibration Blank
ICSA	Final Spectral Interference Check (every 8 hours or less)
ICSAB	Final Spectral Interference Check (every 8 hours or less)

18.0 Revision History

- Revision 10, dated 29 February 2008
  - Integration for TestAmerica and STL operations.
  - Addition of control limits for ICSA and ICSAB.
  - Addition of text on Spectral Interference Check Solutions
  - Change from CRDL/CRQL to CRL, current terminology
  - Addition of current catalog numbers for Table 7 individual elements.
  - Correction of order of Table 9
- Revision 11, dated 17 October 2008
  - Updated to SW-846 Update IV
- Revision 12, dated 30 October 2009
  - Inserted information on new ICV standard, new standards for ICP4.
  - Modified analytical sequence and added ICP4 sequence.
  - Reformatting and condensing.
  - Addition of requirements of Corporate Quality Memorandum CA-Q-QM-004, Technical Guidance on Checking for Spectral Interferences in Optical ICP Analysis, September 24, 2009.
  - Corporate review.

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Title: TOTAL AND AMENABLE CYANIDE (AUTOMATED COLORIMETRIC) WITH OFF-LINE DISTILLATION Method 9012A/B

Approvals (Signature/Date):

Ronald Martin — 6-24-10
Department Supervisor — Date

Michael H. Dunn — 6-24-10
Technical Director — Date

Jared Knierim — 6-25-10
Health & Safety Manager / Coordinator — Date

Eric S. Smith — 6/28/10
Quality Assurance Director — Date

Johnny A. Mitchell — 6.25.10
Laboratory Director — Date

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1.0 Scope and Application

1.1 Analyte, Matrices: This method is used to determine the concentration of inorganic cyanide in groundwater, soils, wastes, or leachate. The method detects inorganic cyanides that are present as either soluble salts or complexes. It is used to determine values for both total cyanide and cyanide amenable to chlorination.

1.2 Reporting Limits: The reporting limit (RL) for water is 0.005 mg/L, for soil, 2.0 mg/kg.

1.3 If for any reason a part of this method cannot be followed, seek the guidance of the Department Supervisor or the Laboratory Technical Director. All abnormalities must be noted on the data or the benchsheet and in the Laboratory Information Management System (LIMS).

2.0 Summary of Method

2.1 The cyanide, as hydrocyanic acid (HCN), is released from samples containing cyanide by means of a reflux-distillation operation under acidic conditions and absorbed in a scrubber containing sodium hydroxide solution. The cyanide ion in the absorbing solution is then determined by automated UV colorimetry.

2.2 In the automated colorimetric measurement, the cyanide is converted to cyanogen chloride (CNCl) by reaction with Chloramine-T at a pH less than 8 without hydrolyzing to the cyanate. After the reaction is complete, color is formed on the addition of pyridine-barbituric acid reagent. The concentration of NaOH must be the same in the standards, the scrubber solutions, and any dilution of the original scrubber solution to obtain colors of comparable intensity.

3.0 Definitions

See TestAmerica Nashville's Quality Assurance Manual Appendix 5 for laboratory definitions. Also, refer to Controlled Document QAF-45, TestAmerica Nashville Acronyms, Keywords, and Definitions.

4.0 Interferences

4.1 Interferences are eliminated or reduced by using the distillation procedure.

4.2 Oxidizing agents such as chlorine decompose most cyanides. Chlorine interferences are removed by adding an excess of sodium arsenite to the waste prior to preservation and storage of the sample to reduce the chlorine to chloride which does not interfere.

4.3 Sulfide interference can be removed by adding an excess of bismuth nitrate to the waste (to precipitate the sulfide) before distillation.

4.4 High results may be obtained for samples that contain nitrate and/or nitrite. During the distillation, nitrate and nitrite form nitrous acid, which react with some organic compounds to form oximes. Once formed, these compounds decompose under test conditions to generate HCN. The possibility of interference of nitrate and nitrite is eliminated by pretreatment with sulfamic acid before distillation.

4.5 Magnesium chloride use accelerates and frees up the cyanide ion.

5.0 Safety

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual and this document. This procedure may involve hazardous material, operations and equipment. This method does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.1 Specific Safety Concerns or Requirements: Strong acids and bases.

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5.2 Primary Materials Used: The following is a list of the materials used in this method, which have a serious or significant hazard rating. **Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material[1]	Hazards	Exposure Limit[2]	Signs and symptoms of exposure
Sulfuric acid	Corrosive Oxidizer Dehydrator	1 mg/m^3	This material causes burns if it comes into contact with the skin or eyes. Inhalation of vapors causes irritation of the nasal and respiratory system.
Sodium hydroxide	Corrosive Poison	2 ppm, 5 mg/m^3	This material causes burns if it comes into contact with the skin or eyes. Inhalation of Sodium Hydroxide dust causes irritation of the nasal and respiratory system.
Chloramine T hydrate	Poison		May be harmful by inhalation, ingestion, or skin absorption. This material is irritating to mucous membranes and upper respiratory tract. Avoid contact and inhalation.
Pyridine	Flammable Irritant	5 ppm-TWA	Inhalation causes severe irritation to the respiratory tract. Symptoms of overexposure include headache, dizziness, nausea, and shortness of breath. Causes severe irritation possibly burns, to the skin. Symptoms include redness and severe pain. Absorption through the skin may occur, resulting in toxic effects similar to inhalation. May act as a photosensitizer. Vapors cause eye irritation. Splashes cause severe irritation, possible corneal burns and eye damage.
Potassium cyanide	Poison Corrosive	5 mg/m^3 TWA as CN	**This material forms Hydrogen Cyanide (HCN) gas when combined with strong acids. Breathing HCN gas may result in death.** Corrosive to the respiratory tract. May cause headache, weakness, dizziness, labored breathing nausea and vomiting, which can be followed by weak and irregular heart beat, unconsciousness, convulsions, coma and death. Solutions are corrosive to the skin and eyes, and may cause deep ulcers, which heal slowly. May be absorbed through the skin, with symptoms similar to those noted for inhalation. Symptoms may include redness, pain, blurred vision, and eye damage.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			
TWA = Time-weighted average			

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6.0 Equipment and Supplies

6.1 Instrumentation

- Automated continuous-flow analytical instrument (i. e., Lachat Model 8000 or 8500) with autosampler, cyanide manifold, and peristaltic pump, colorimeter with a 1 cm flowcell and 570 ηm filter, Omnion data acquisition system or Discrete Analyzer (i. e., Konelab).
- Distillation system, Andrews Glass Midi-Dist, or equivalent.
- pH Meter
- Vacuum pump
- Balance, 0-160 gram capacity.

6.2 Supplies

- 100- and 250-mL volumetric flasks, Class A.
- Class A pipets.
- Sea or Ottawa sand for blank soil matrix and LCS soil matrix.
- Plastic, certified, centrifuge tubes.

7.0 Reagents and Standards

7.1 Reagent water, analyte-free, ASTM Type II or equivalent.

7.2 Reagents for sample collection, preservation, and handling

7.2.1 Sodium arsenite (0.1 N), $NaAsO_2$. Dissolve 3.2 g $NaAsO_2$ in 250 mL reagent water.

7.2.2 Sodium hydroxide solution (10N), NaOH. Commercially available, or 400 g NaOH/L reagent water.

7.2.3 Acetic acid (1.6 M) CH_3COOH. Dilute one part of concentrated acetic acid with 9 parts of water.

7.3 Reagents for cyanides amenable to chlorination

7.3.1 Calcium hypochlorite solution: Dissolve 5 g of calcium hypochlorite ($Ca(OCl)_2$) in 100 mL of reagent water.

7.3.2 Sodium Hypochlorite: Commercial Bleach (No additives or scents), 5 to 6% hypochlorite.

7.3.3 Sodium hydroxide solution (0.25 N), NaOH. Dissolve 10 g of NaOH in 1 liter of reagent water.

7.3.4 Ascorbic acid crystals, commercial source.

7.3.5 Potassium iodide starch paper, commercial.

7.4 Reagents for distillation

7.4.1 Sodium hydroxide (0.25 N). Dissolve 10 g NaOH in 1 liter reagent water.

7.4.2 Bismuth nitrate (0.062 M), $Bi(NO)_3*5H_2O$. Dissolve 30 g $Bi(NO)_3*5H_2O$ in 100 mL of reagent water. While stirring, add 250 mL of glacial acetic acid, CH_3COOH. Stir until dissolved and dilute to 1 liter with reagent water.

7.4.3 Sulfamic acid, H_2NSO_3H. Dissolve 200 g H_2NSO_3H in 1 liter of reagent water.

7.4.4 Sulfuric acid (18 N), H_2SO_4. Slowly and carefully add 500 mL of concentrated H_2SO_4 to 500 mL of reagent water or use a commercially prepared solution.

7.4.5 Magnesium chloride solution (2.5 M), $MgCl_2*6H_2O$. Dissolve 510 g of $MgCl_2*6H_2O$ in 1 liter of reagent water.

7.4.6 Lead acetate paper, commercial.

7.5 Reagents for automated colorimetric determination (Lachat)

7.5.1 Pyridine-barbituric acid reagent: Place 15 g of barbituric acid in a 1-liter volumetric flask, add 100 mL reagent water, rinsing down the sides of the flask to wet the barbituric acid. Add 75 mL of pyridine and mix. Add 15 mL of concentrated HCl, mix and cool to room temperature. Dilute to 1 liter with reagent water and mix. This reagent is stable for approximately six months if stored in a cool, dark place.

7.5.2 Chloramine-T solution: Dissolve 2.0 g of white, water-soluble chloramine-T in 500 mL of reagent water. Prepare fresh daily.

7.5.3 Sodium hydroxide, 10 N: Commercial source.

7.5.4 Carrier: 0.25 N NaOH.

7.5.5 Buffer: In a 1-liter volumetric flask, dissolve 97 g of potassium phosphate, monobasic anhydrous, (KH_2PO_4) in approximately 800 mL reagent water. Dilute to volume and invert to mix. Prepare fresh monthly.

7.5.6 All working standards must be prepared in 0.25 N NaOH.

7.5.7 Dilution water and receptacle wash water (NaOH, 0.25 N): Dissolve 10.0 g NaOH in 500 mL of reagent water. Dilute to 1 liter.

7.6 Stock cyanide solution: Dissolve 2.51 g of KCN and 2 g KOH in 900 mL of reagent water and mix. Dilute to one liter. Standardize with 0.0192 N $AgNO_3$ to appropriate concentration. 1 mL = 1 mg CN. Purchased standards may be used in place of prepared solutions. Dilute to make a 10 µg/mL working standard. Standardize stock against silver nitrate weekly.

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7.7 Reagents for automated colorimetric determination (Konelab): See Konelab Methods Manual.

7.8 See SOP Reagent and Standard Purchase, Preparation, Control, Documentation / NV08-214 for shelf-life and storage requirements for standards and reagents. Also, refer to benchsheets, logbooks, and LIMS.

8.0 Sample Collection, Preservation, Shipment and Storage

Matrix	Sample Container	Min. Sample Size	Preservation	Holding Time	Reference
Water	HDPE or Glass	250 mL	Cool 0-6°C, Preserve with 4 mL 10 N NaOH/L sample at the time of collection (pH ≥ 12)	14 days from collection to analysis	SW-846 Section 2.0
Soil	HDPE or Glass	30 g	Cool 0-6°C		

- All bottles must be thoroughly cleansed and rinsed to remove soluble material from containers. Certified clean containers are acceptable.
- Oxidizing agents such as chlorine decompose most of the cyanides. To determine whether oxidizing agents are present, test a drop of the sample with potassium iodide-starch test paper (KI starch paper); a blue color indicates the need for treatment. Add 0.1 N sodium arsenite a few drops at a time until a drop of sample produces no color on the indicator paper. Add an additional 5 mL of sodium arsenite for each liter of sample volume.

9.0 Quality Control

The laboratory maintains a formal quality assurance program and records to document the quality of the data generated.

9.1 Sample QC

The following quality control samples are prepared with **each batch of 20 or fewer samples, except for samples from OK and WY (10 samples/batch)**.

Quality Controls	Acceptance Criteria		Corrective Action
	Water	Soil	
Method Blank	< RL		Correct problem then re-prep and analyze blank and all samples processed with the contaminated blank. If target >10x blank, report but qualify.
Laboratory Control Sample (LCS)[1], second source, distilled	90-110% recovery	80-120% recovery	If %R is low, redistill, rerun. If high, report non-detect samples, redistill and reanalyze any positive samples.
Matrix Spike	See CLM for historical % recovery range.		Report. Qualify as needed.
Matrix Spike Duplicate	See CLM for historical % recovery range.		Report. Qualify as needed.
Sample Duplicate	See CLM for historical maximum RPD.		Report. Qualify as needed.

CLM = Control Limits Manual (contains applicable current control limits).

[1]For AZ, TX, WV samples, a LCS duplicate is required.

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- The **Method blank:** add method reagents to 50 mL reagent water (for water batches) or to 1 gram sea sand and 50 mL reagent water (for soil batches).
- **Laboratory Control Sample (LCS)**: A **distilled LCS** is run with each batch.
  - Prepare the LCS (0.1 µg/mL) by adding 50 µL of 2nd source 100 µg/mL cyanide standard to 50 mL reagent water (for water batches) or to 1 gram sea sand and 50 mL reagent water (for soil batches).
- **Matrix Spike / Matrix Spike Duplicate**: Both the matrix spike and matrix spike duplicate are brought through the entire sample preparation and analytical process.
  - A matrix spike is prepared by adding cyanide from the working standard standard to 50 mL or 1 gram of sample to ensure a concentration of approximately 100 µg/L (using 50 µL of 100 µg/mL cyanide stock standard to 10 mL 0.25N NaOH).
- **Sample Duplicate:** A second aliquot of one sample in the batch is distilled and analyzed as an indication of precision.

9.2 Instrument QC

Quality Controls	Frequency	Acceptance Criteria	Corrective Action
Initial calibration (minimum six standards)	Initial calibration prior to sample analysis.	$r^2 \geq 0.99$, $r \geq 0.995$ for first-order linear regression	Correct problem then repeat initial calibration
Initial Calibration Verification Sample (ICV), second source, not distilled	Immediately after calibration	90-110% recovery	Re-calibrate
Initial Calibration Blank (ICB)	Immediately after the ICV	< RL	Correct the problem and re-run.
Continuing Calibration Verification Sample (CCV), distilled & non-distilled	Distilled: 1/batch; Non-distilled: every 10 samples	90-110% recovery	Correct, re-calibrate, rerun samples. If high and samples are ND, it is acceptable to report the results.
Continuing Calibration Blank (CCB)	After each CCV	< RL	Correct problem, repeat samples.

- **Initial Calibration Standards**: See Section 10.2.
- **Initial Calibration Verification (ICV)**
  - Prepare a 0.2 µg/mL ICV by adding 100 µL of 100 µg/mL 2nd source standard to 50.0 mL reagent water.
- **Continuing Calibration Verification Sample (CCV)**
  - Use 100 µL 100 µg/mL cyanide standard used for calibration to 50 mL reagent water for a final concentration of 0.2 µg/mL.
- **Initial and Continuing Calibration Blank (ICB and CCB)**: Use 0.25N NaOH.

10.0 Procedure

10.1 Sample Preparation

- **Sample size**

Matrix	Sample Size
Water	50 mL
Soil	1 gram

- **Pretreatment for cyanides amenable to chlorination**

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- This test **must** be performed in the hood, covering all containers with aluminum foil. $K_3[Fe\text{-}(CN)_6]$ may decompose under UV light and hence test positive for cyanide amenable to chlorination if exposed to fluorescent lighting or sunlight. Two identical sample aliquots are required to determine cyanides amenable to chlorination.

1	To a 50.0 mL aliquot of sample, add bleach or calcium hypochlorite solution solution dropwise while agitating and maintaining the pH between 11 and 12 with 1.25 N sodium hydroxide until an excess of chlorine is present as indicated by KI-starch paper turning blue. The sample is subjected to alkaline chlorination by this step. Designate this aliquot "AM." CAUTION: The initial reaction product of alkaline chlorination is the very toxic gas cyanogen chloride; therefore, it is necessary that this reaction be performed in a hood.
2	Test for excess chlorine with KI-starch paper and maintain this excess for one hour with continuous agitation. A distinct blue color on the test paper indicates a sufficient chlorine level. If necessary, add additional bleach solution.
3	After one hour, add ascorbic acid until KI-starch paper shows no residual chlorine. Add an excess ascorbic acid to ensure the presence of excess reducing agent.
4	Test for total cyanide as described below in both the chlorinated and the unchlorinated samples. The difference of total cyanide in the chlorinated and unchlorinated samples is the cyanide amenable to chlorination.
5	Prepare an amenable blank using reagent water and an amenable LCS (0.1 µg/mL) with the samples for chlorination, and run a regular total blank and a total LCS with the run for verification.

- **Distillation Procedure**

1	Use lead acetate paper to check for sulfide. A positive result is indicated by a black color. Add bismuth nitrate, mix, and retest. Once sulfide is ND, transfer the sample to the distillation vessel. Add additional 2 mL bismuth nitrate solution through the air inlet tube. Connect the distillation vessel, condenser, gas scrubber and vacuum trap.
2	Start a slow stream of air entering the distillation vessel by adjusting the vacuum valve. Adjust the vacuum so that approximately two bubbles of air per second enter the boiling flask through the air inlet tube.
3	Add 2 mL of sulfamic acid solution through the air inlet tube. Mix for three minutes. **Keep bismuth nitrate to sulfamic acid ratio to 1:1.** Note: Excessive use of sulfamic acid could create method bias.
4	Slowly add 5 mL of 18 N sulfuric acid through the air inlet tube. Rinse the tube with water and allow the airflow to mix the flask contents for three minutes. Add 2 mL of 2.5 M magnesium chloride through the air inlet and wash the inlet tube with a stream of water.
5	Set the timer to 90 minutes (30 minutes to heat up and 60 minutes at temperature). Continue the airflow for at least 15 minutes after the 60 minutes. After cooling the boiling flask, and closing the vacuum source, disconnect the gas scrubber.
6	Transfer the solution from the scrubber into a plastic, certified, centrifuge tube and analyze on the Lachat or Konelab.

10.2 Calibration: Refer to SOP Selection of Calibration Points / CA-P-T-002. See Section 11 for equations. Calculations are performed by vendor software and LIMS.

1	Prepare a series of standards by pipetting suitable volumes of working standard potassium

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	cyanide solution in 0.25 N NaOH with a final volume of each standard being 10.0 mL. The low standard must be at or below the reporting limit (RL).

mL of 10.0 µg/mL Standard Solution	Final Volume (mL) with 0.25 N NaOH	Concentration (µg/mL)
0.004 mL	10.0	0.004
0.01 mL	10.0	0.01
0.02 mL	10.0	0.02
0.04 mL	10.0	0.04
0.1 mL	10.0	0.1
0.2 mL	10.0	0.2
0.4 mL	10.0	0.4

2	Place calibration standards in test tubes or cuvettes and start method. Obtain absorbance values for the standard curve.
3	Prepare a standard curve by plotting absorbance of standard versus the cyanide concentration. The correlation coefficient (r^2) must be ≥ 0.99 (r ≥ 0.995) or re-calibrate. See Section 11 for applicable equations.
4	**It is required that at least two standards (a high and a low) be distilled and compared to similar values on the curve to ensure that the distillation technique is reliable. If distilled standards do not agree within ± 10% of the undistilled standards, the analyst must find the cause of the apparent error before proceeding.**

10.3 Automated colorimetric determination (Konelab): Use settings in this table; see SOP Konelab / NV07-141.

Full Name	Total Cyanide				
Online Name		Test In Use	YES		
Test type	Photometric		LOW	HIGH	
		Test limit	0.000	4000.000 µg/L	
Result unit	µg/L	Initial absorbance	-0.010	2.500 A	
Number of Decim.	3	Dilution limit	*	400.0000 µg/L	
		Secondary dil 1+	0.0	9.0	
		Critical limit	*	*	
		Reflex test limit	*	*	µg/L
		Reflex test			
Acceptance	Manual	Reference class	LOW	HIGH	In Use
Dilution 1+	0.0				
Sample type	Water	Correction factor	1.00		
	Raw water	Correction bias	0.00	µg/L	
	Sewage				
Calibration type	Linear				
Repeat time (d)	0	Abs error (mA)	*		
Points/cal.	Single	Rel error (%)	*		
Acceptance	Manual				

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Response limit (mA)	MIN	MAX			
	*	*			
Bias correction in use	NO				
Cd reduction	NO				
Type of Calibrators	Series				
Calibrator	Conc.	Dil. Ratio			
Cn-400	400.000	1+99.0			
Cn-400	400.000	1+39.0			
Cn-400	400.000	1+19.0			
Cn-400	400.000	1+9.0			
Cn-400	400.000	1+99.0			
Cn-400	400.000	1+3.0			
Cn-400	400.000	1+1.0			
Cn-400	400.000	1+0.0			
Manual QC in Use	YES	Routine QC in Use	YES		
Acceptance	Manual	Interval requests		10	
		Additional condition	NO		
Control	Mean	SD	Control	Mean	SD
Cn-BLANK	0.00	3.00	Cn-BLANK	0.00	3.00
Cn-ICV	100.00	10.00	Cn-CCV	200.00	20.0
Rules in Use		1:1.0 *SD	Rules in Use		1:1.0*SD
Blank		YES	Normal cuvette		
Sample		Volume (µL)		100	
Disp. With		Extra	Add. Volume (µL)	30	
Dilution with		Special	Wash reagent	Water	
Raw Sample					
Disp. With		Extra	Add. Volume (µL)	30	
Special diluent		0.25 NaOH			
Disp. With		Extra	Add. Volume (µL)	10	
Reagent		CN-BUFFER	Volume (µL)	40	
Disp. With		Extra	Add. Volume (µL)	30	
Wash reagent		water			
Reagent wash		Before dispense			
Reagent		CN-CHLOR T	Volume (µL)	4	
Disp. With		Extra	Add. Volume (µL)	30	
Wash reagent		water			
Reagent wash		Before dispense			
Incubation			Time (sec)	60	
Measurement		End point	Blank		

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Resp. Min (A)		-0.010	Resp. Max (A)	*	
Reagent		CY-PYR BAR	Volume (µL)	20	
Disp. with		Water	Add. Volume (µL)	30	
Wash reagent		water			
Reagent wash		Before dispense			
Incubation			Time (sec)	480	
Measurement		End point			
Wavelength (nm)		575 nm	Side wavel. (nm)	None	
Meas. Type		Fixed timing			

10.4 Automated Colorimetric Determination for Lachat Auto-analyzer

1	Set up the Lachat auto analyzer (See SOP Lachat / NV07-39).
2	Allow colorimeter and recorder to warm up. Run a baseline with all reagents, feeding reagent water through the sample line.
3	Place appropriate standards in the sampler in order of decreasing concentration. Complete loading of the sampler tray with unknown samples.
4	When the baseline becomes steady, begin the analysis.

10.5 Example Analysis Queue / Sequence

1	Method Blank
2	ICV, 2nd source, not distilled
3	ICB
4	CCV, 1° source, not distilled
5	CCB
6	LCS, 2nd source, distilled
7	CCV, distilled
8	Sample 1
9	Matrix Spike
10	Matrix Spike Duplicate
11	Samples 2-10
12	CCV, not distilled
12	CCB
13	Samples 11-20
14	Sample Duplicate
15	CCV, 1° source
16	CCB

11.0 Calculations / Data Reduction

11.1 Accuracy

$$\text{LCS \% Recovery} = \frac{\text{Measured concentration}}{\text{Known concentration}} \times 100$$

11.2 Precision (RPD)

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RPD = Absolute value (orig. MS value - dup. MS value) x 100
(Orig. MS value + dup. MS value)/2

11.3 Linear calibration using a least squares regression: A linear calibration model based on a least squares regression of instrument response versus the concentration of the analyte is employed. Ensure that the instrument response is treated as the dependent variable (y) and the amount as the independent variable (x). This is a statistical requirement and is not simply a graphical convention.

For external standard calibration, x is the mass of the analyte in the sample aliquot introduced into the instrument, and y is the area of the response, as in:

$x = C_s$ and $y = A_x$

A linear least squares regression constructs a linear equations of the form:

$y = ax + b$

By minimizing the differences between the observed results (y_i, the instrument response) and the predicted results (y'_i, the response calculated from the constructed equation). The regression equation is

$y'_i = ax_i + b$

a = Regression coefficient or the slope of the line.
b = the y-intercept.
y'_i = Predicted (or calculated) response for the i^{th} calibration standard.
x_i = concentration of analyte in the i^{th} calibration standard aliquot introduced into the instrument.

The sum of the squares of the differences is minimized to obtain a and b:

$$\sum_{i=1}^{n}(y_i - y'_i)^2$$

where n is the total number of calibration points. The regression calculations attempt to minimize this sum of the squares, hence the name "least squares regression."

Weighting the sum of the squares of the differences may significantly improve the ability of the least squares regression to fit the linear model to the data. The general form of the sum of the squares of the differences containing the weighting factor is:

$$\sum_{i=1}^{n}(y_i - y'_i)^2 w_i$$

w_i = Weighting factor for the i^{th} calibration standard (w = 1 for unweighted least squares regression).
y_i = Observed instrument response (area for the i^{th} calibration standard.

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y'_i = Predicted (or calculated response for the i^{th} calibration standard.
n = total number of calibration standards.

The mathematics used in least squares regression has a tendency to favor numbers of larger value over numbers of smaller value. Thus the regression curves that are generated tend to fit points that are at the upper calibration levels better than those points at the lower calibration levels.

To compensate for this, a weighting factor which reduces this tendency can be used. Examples of weighting factors which can place more emphasis on numbers of smaller value are:

$w_i = 1/y_i$ or $w_i = 1/y_i^2$

There are numerous other ways to define weighting factors, but these are recommended if a weighting factor other than 1 ($w_i = 1$) is to be used.

11.4 Coefficient of Determination **Correlation Coefficient**

$$r^2 = \frac{\left(\sum xy\right)^2}{\sum x^2 \sum y^2} \qquad r = \frac{\left(\sum xy\right)}{\sqrt{\sum x^2 \sum y^2}}$$

y = Response
x = Concentration

11.5 Result Calculation: Concentrations of samples are calculated by the instrument by comparing sample peak response with the calibration curve to determine the concentration:

$$\textbf{Concentration (mg/L or µg/g)} = \frac{\text{(Conc. (µg/mL), instrument) x Dilution X Volume Scrubber (mL)}}{\text{Volume (mL) or Mass (gram)}}$$

Dilution =1 if no dilution

Note: Amenable CN = Total CN-Treated tube reading.

12.0 Method Performance

12.1 Method Detection Limit Study (MDL): The method detection limit (MDL) is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. The MDL is determined according to the laboratory's MDL procedure in SOP Determination of Method Detection Limits / NV08-202. MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix, and may not be achievable in all environmental matrices. The laboratory maintains MDL studies for analyses performed; these are verified at least annually unless method requirements require a greater frequency.

12.2 Demonstration of Capability: The laboratory demonstrates initial proficiency by generating data of acceptable accuracy and precision for target analyses in a clean matrix. The laboratory also repeats the operation whenever new staff is trained or significant changes in instrumentation are made and on an annual basis thereafter. See the training section of TestAmerica Nashville's QA Manual and SOP Training / NV08-199 for information on how to accomplish this demonstration.

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12.3 Training Requirements: Demonstration of Capability is performed initially when learning the method and annually thereafter. Four Laboratory Control Samples resulting in an average % recovery within the control limits and a precision less than the quality control maximum are required.

12.4 Proficiency Testing Studies: The laboratory participates in formal proficiency testing (PT) studies, where solutions of unknown concentrations are analyzed and the performance of all participants is compared. See the QA department for the results of recent PT studies.

13.0 Pollution Control

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i. e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention."

14.0 Waste Management

14.1 Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes must be stored, managed, and disposed of in accordance with all federal and state laws and regulations. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to the QA Manual and SOP Waste Disposal / NV10-83.

14.2 Wastestreams Produced by the Method:

- Aqueous alkaline material from the auto-analyzer/titrations is disposed of in the hazardous waste, toxic, corrosive drum in the waste disposal area.

15.0 References / Cross-References

15.1 EPA Method 9012A, SW-846 Update III Revision 1, December 1996; 9010B and 9012B, SW-846 Update IIIB Revision 2, November 2004.
15.2 Lachat Method 10-204-001-A.
15.3 Konelab Methods and Instrument Manuals.
15.4 TestAmerica Nashville's Quality Assurance Manual.
15.5 TestAmerica Nashville's Control Limits Manual.
15.6 Corporate Environmental Health and Safety Manual (CW-E-M-001)
15.7 SOPs: Selection of Calibration Points / CA-P-T-002, Waste Disposal / NV10-83, Training Procedures for Environmental Technical Staff / NV08-199, Determination of Method Detection Limits / NV08-202, Lachat / NV07-39, Konelab / NV07-141, Reagent and Standard Purchase, Preparation, Control, Documentation / NV08-214.
15.8 Controlled Document: QAF-45, TestAmerica Nashville – Acronyms, Keywords, and Definitions.

16.0 Method Modifications

Item	Modification
1	Treatment for samples with sulfide interference
2	Lachat Auto-analyzer
3	Konelab Automated Analyzer

17.0 Attachment

None.

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18.0 Revision History

- Revision 7, 30 June 2008
  - Integration for TestAmerica and STL operations.
- Revision 8, 23 October 2009
  - Addition of preparation of prep blank.
  - Addition of OH VAP statements for prep blank and LCS.
  - Addition of Corporate Environmental Health and Safety Manual, Lachat, Konelab, and Standard/Reagent SOPs to reference section.
- Revision 9, 29 June 2010
  - Removal of reference to EPA 335.2 CLP-M.
  - Addition of Section 14.2, QAF-45, and the use of weighted calibration curves.

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TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

Nashville
Standard Operating Procedure
(SOP) Change Form

SOP Number/Revision No.: **8270 / NV/SA04-22.13** Effective Date: **11/30/10**
Last Mod. Date: 10/9/09

SOP Title: **Method 8270C/D: Semivolatile Organic Compounds by Gas Chromatography / Mass Spectrometry (GC/MS)**

Affected SOP Section Number(s): Sections 7.3 and 9.1

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ISSUED TO: QA Server, QA, 04B

Revision Number with Mod ID: **13a**

The following SOP change is in effect as of the stated date. This form will remain attached to the referenced SOP until such a time that the SOP is updated, approved, and redistributed, at which time it will become part of the historical SOP record. ***Append this form to the front of the SOP copy.***

1. Reason for SOP Change:

☐ Typographical Corrections (Non-Technical) – Re-Training Not Required.

■ Typographical Corrections (Technical – Define) – Analyst acknowledgement of corrections is required.

■ Procedural Changes (Define Below) – Re-Training Required.

☐ Other

2. Summary of Procedure Change

Section 7.3, first bullet: Change CUS-5160 to CUS-6150.

Section 9.1, Laboratory Control Sample bullet, second sentence from the end of the paragraph: Add "target" to: "All target analytes must meet the LCS QC criteria . . . .

	11/29/10		11-26-10
Supervisor Approval	Date	Technical Director Approval	Date
	11/29/10		11-26-10
Quality Assurance Approval	Date	Laboratory Director Approval	Date

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Title: SEMIVOLATILE ORGANIC COMPOUNDS BY GAS CHROMATOGRAPHY/MASS SPECTROMETRY (GC/MS) EPA Method 8270C/D

Approvals (Signature/Date):

Cory Spry 10/1/09
Department Supervisor — Date

Michael H. Dunn 9.26.09
Technical Director — Date

Jared Knierim 10-2-09
Health & Safety Manager / Coordinator — Date

Eric S. Smith 9/25/09
Quality Assurance Director — Date

Johnny A. Mitchell 10-6-09
Laboratory Director — Date

For NELAP purposes, this SOP is known as 8270 / **SA**04-22. The new NV designation derives from the integration of TestAmerica and STL.

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Facility Distribution No. 04-22 **Distributed To:** QA Server, QA, 04B

1.0 Scope and Application

1.1 Analyte, Matrices: This method is used to determine the concentration of semivolatile organic compounds in extracts prepared from many types of oily wastes, soils/sediments, and water samples. The following compounds can be determined by this method:

Analyte	CAS #	Analyte	CAS #
Acenaphthene[1, 2, 5]	83-32-9	Hexachlorocyclopentadiene[1, 2]	77-47-4
Acenaphthene-d_{10} (IS)		Hexachloroethane[1, 2]	67-72-1
Acenaphthylene[1, 2, 5]	208-96-8	Hexachlorophene[2]	70-30-4
Acetophenone[2]	98-86-2	Hexachloropropene[2]	1888-71-7
2-Acetylaminofluorene[2]	53-96-3	Indeno(1,2,3-cd)pyrene[1, 2, 5]	193-39-5
4-Aminobiphenyl[2]	92-67-1	*Indene*[4]	
Aniline[2]	62-53-3	Isodrin[2]	465-73-6
Anthracene[1, 2, 4, 5]	120-12-7	Isophorone[1, 2]	78-59-1
Aramite[2]	140-57-8	*cis-Isosafrole*[2]	17627-76-8
Azobenzene[3]	103-33-3	*trans-Isosafrole*[2]	4043-71-4
Benzidine[3]	92-87-5	Kepone[2]	143-50-0
Benzoic acid[3]	65-85-0	Methapyrilene[2]	91-80-5
Benz(a)anthracene[1, 2, 4, 5]	56-55-3	3-Methylcholanthrene[2]	56-49-5
Benzo(b)fluoranthene[1, 2, 4, 5]	205-99-2	*6-Methyl chrysene*[4]	1705-85-7
Benzo(j)fluoranthene[4]		4,4'-Methylenebis(2-chloroaniline) (MOCA)[3]	101-14-4
Benzo(k)fluoranthene[1, 2, 4, 5]	207-08-9	Methyl methanesulfonate[2]	66-27-3
Benzo(g,h,i)perylene[1, 2, 5]	191-24-2	*1-Methylnaphthalene*[3, 4, 5]	90-12-0
Benzo(a)pyrene[1, 2, 4, 5]	50-32-8	2-Methylnaphthalene[1, 2, 5]	91-57-6
Benzyl alcohol[2]	100-51-6	Methyl parathion[2]	298-00-0
Bis(2-chloroethoxy)methane[1, 2]	111-91-1	2-Methylphenol[1, 2, 4]	95-48-7
Bis(2-chloroethyl)ether[1, 2]	111-44-4	3-Methylphenol[1, 2, 4]	108-39-4
Bis(2-chloroisopropyl)ether[1, 2]	108-60-1	4-Methylphenol[1, 2, 4]	106-44-5
Bis(2-ethylhexyl)adipate[3]	103-23-1	Naphthalene[1, 2, 4, 5]	91-20-3
Bis(2-ethylhexyl)phthalate[1, 2, 4]	117-81-7	Naphthalene-d_8 (IS)	
Bisphenol A[3]	80-05-7	1,4-Naphthoquinone[2]	130-15-4
4-Bromophenyl phenylether[1, 2]	101-55-3	1-Naphthylamine[2]	134-32-7
Butyl benzyl phthalate[1, 2, 4]	85-68-7	2-Naphthylamine[2]	91-59-8
Carbazole[1]	86-74-8	2-Nitroaniline[1, 2]	88-74-4
4-Chloroaniline[1, 2]	106-47-8	3-Nitroaniline[1, 2]	99-09-2
Chlorobenzilate[2]	510-15-6	4-Nitroaniline[1, 2]	100-01-6
4-Chloro-3-methylphenol[1, 2]	59-50-7	Nitrobenzene[1, 2]	98-95-3
1-Chloronaphthalene[3]	90-13-1	Nitrobenzene-d_5 (surr)	
2-Chloronaphthalene[1, 2]	91-58-7	2-Nitrophenol[1]	88-75-5
2-Chlorophenol[1, 2]	95-57-8	4-Nitrophenol[1, 2]	100-02-7
2-Chlorophenol-d_4 (surr)		5-Nitro-o-toluidine[2]	99-55-8
4-Chlorophenyl phenylether[2]	7005-72-3	Nitroquinoline-1-oxide[2]	56-57-5
Chrysene[1, 2, 4, 5]	218-01-9	n-Nitrosodi-n-butylamine[2]	924-16-3
Chrysene-d_{12} (IS)		n-Nitrosodiethylamine[2]	55-18-5
n-Decane[3]	124-18-5	n-Nitrosodimethylamine[2]	62-75-9
Diallate (cis and trans)[2]	2303-16-4	n-Nitrosomethylethylamine[2]	10595-95-6
Dibenz(a,h)acridine[4]	226-36-8	n-Nitrosodiphenylamine[1, 2] and Diphenylamine	86-30-6 and 122-39-4
Dibenz(a,j)acridine[3]	224-42-0	n-Nitrosodi-n-propylamine[1, 2]	621-64-7
Dibenz(a,h)anthracene[1, 2, 4, 5]	53-70-3	n-Nitrosomorpholine[2]	59-89-2
Dibenzofuran[1, 2]	132-64-9	n-Nitrosopiperidine[2]	100-75-4
2,3-Dichloroaniline[3]	608-27-5	n-Nitrosopyrrolidine[2]	930-55-2

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1,2-Dichlorobenzene[1, 2, 4]	955-50-1	*n-Octadecane*[3]	593-45-3
1,2-Dichlorobenzene-d_4(surr)		Parathion[2]	56-38-2
1,3-Dichlorobenzene[1, 2, 4]	541-73-1	Pentachlorobenzene[2]	608-93-5
1,4-Dichlorobenzene[1, 2, 4]	106-46-7	*Pentachloroethane*[2]	76-01-7
1,4-Dichlorobenzene-d_4(IS)		Pentachloronitrobenzene[2]	82-68-8
3,3'-Dichlorobenzidine[1, 2]	91-94-1	Pentachlorophenol[1, 2]	87-86-5
2,4-Dichlorophenol[1, 2]	120-83-2	Perylene-d_{12} (IS)	
2,6-Dichlorophenol[2]	87-65-0	Phenacetin[2]	62-44-2
Diethyl phthalate[1, 2, 4]	84-66-2	Phenanthrene[1, 2, 4, 5]	85-01-8
Dimethoate[2]	60-51-5	Phenanthrene-d_{10} (IS)	
Dimethylaminoazobenzene[2]	60-11-7	Phenol[1, 2, 4]	108-95-2
7,12-Dimethylbenz(a)anthracene[2, 4]	57-97-6	Phenol-d_5 (surr)	
3,3'-Dimethylbenzidine[2]	119-93-7	1,4-Phenylenediamine[2]	106-50-3
2,4-Dimethylphenol[1, 2, 4]	105-67-9	Phorate[2]	298-02-2
a,a- Dimethylphenethylamine[2]	122-09-8	2-Picoline (2-Methylpyridine)[2]	109-06-8
Dimethyl phthalate[1, 2, 4]	131-11-3	Pronamide[2]	23950-58-5
Di-n-butyl phthalate[1, 2, 4]	84-74-2	Pyrene[1, 2, 4, 5]	129-00-0
1,3-Dinitrobenzene[2]	99-65-0	Pyridine[2, 4]	110-86-1
4,6-Dinitro-2-methylphenol[1, 2]	534-52-1	*Quinoline*[4]	91-22-5
2,4-Dinitrophenol[1, 2, 4]	51-28-5	Safrol[2]	94-59-7
2,4-Dinitrotoluene[1, 2, 5]	121-14-2	Terphenyl-d_{14}(surr)	1718-51-0
2,6-Dinitrotoluene[1, 2, 5]	606-20-2	*Alpha-Terpineol*[3]	7785-53-7
Dinoseb[2]	88-85-7	1,2,4,5-Tetrachlorobenzene[2]	95-94-3
1,4-Dioxane	123-91-9	2,3,4,6-Tetrachlorophenol[2]	58-90-2
1,2-Diphenylhydrazine[3]	122-66-7	*Tetraethyl dithiopyrophosphate (Sulfotepp)*[2]	3689-24-5
Di-n-octyl phthalate[1, 2, 4]	117-84-0	Tetraethylpyrophosphate[3]	107-49-3
Disulfoton[2]	298-04-4	Thionazine[2]	297-97-2
Ethyl methanesulfonate[2]	62-50-0	Thiophenol[4]	108-98-5
Famphur[3]	52-85-7	o-Toluidine[2]	95-53-4
Fluoranthene[1, 2, 4, 5]	206-44-0	2,4,6-Tribromophenol (surr)	118-79-6
Fluorene[1, 2, 5]	86-73-7	1,2,4-Trichlorobenzene[1, 2]	120-82-1
2-Fluorobiphenyl(surr)	321-60-8	2,4,5-Trichlorophenol[1, 2]	95-95-4
2-Fluorophenol (surr)	367-12-4	2,4,6-Trichlorophenol[1, 2]	88-06-2
Hexachlorobenzene[1, 2]	118-74-1	o,o,o-Triethylphosphorothioate[2]	126-68-1
Hexachlorobutadiene[1]	87-68-3	1,3,5-Trinitrobenzene[2]	99-35-4
Compounds in italics are not present in the EPA method.			
[1] – Normal laboratory 8270 compounds. ***For OH VAP projects, report only compounds designated by a 1 superscript; see Attachment 5.***			
[2] - Appendix IX compounds (by request only)			
[3] - additional compounds available by this method (by request only)			
[4] - Skinner List for Refinery Waste compounds (by request only)			
[5] - Compounds that are available by GC/MS-SIM (by request only)			
IS = These compounds are used as internal standards.			
surr = These compounds are used as surrogates.			

This method is used to quantitate neutral, acidic, and basic organic compounds that are soluble in methylene chloride and capable of being eluted, without derivatization, from a gas chromatographic fused-silica capillary column coated with a slightly polar methyl silicone phase. This method is not appropriate for the quantitation of multi-component analytes, e. g., Aroclors, Toxaphene, Chlordane, etc., because of limited sensitivity for those analyses. This method is appropriate for confirmation of the presence of these analytes when concentration in the extract

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permits. However, it is appropriate for the multi-component analyte, Diesel Range Organics (DRO), as requested by Missouri and California; see Attachment 6.

1.2 Reporting Limits: The laboratory typical report limit (RL) is approximately 2 - 100 µg/L for water samples, 67 - 670 µg/kg (wet weight) for soil/sediment samples, and 10 - 1000 mg/kg for wastes (dependent on matrix and method of preparation). See the following table for typical RLs for each compound. For the most current analyte RLs, refer to the current Control Limits Manual (CLM) or LIMS.

Typical Reporting Limits for 8270 Compounds

Analyte	Water RL µg/L	Soil RL mg/kg	Analyte	Water RL µg/L	Soil RL mg/kg
♦**Acenaphthene**	10	0.333	♦*Isosafrole*	50	1.67
♦**Acenaphthylene**	10	0.333	♦*Kepone*	10	0.333
♦*Acetophenone*	10	0.333	♦*Methapyrilene*	50	0.333
♦*2-Acetylaminofluorene*	10	0.333	♦*3-Methylcholanthrene*	10	0.333
♦*4-Aminobiphenyl*	10	1.67	♣*6-Methylchrysene*	10	0.333
♦*Aniline*	50	1.67	♦*Methyl methanesulfonate*	10	0.333
♦♣**Anthracene**	10	0.333	♣*1-Methylnaphthalene*	10	0.333
♦*Aramite*	50	1.67	♦**2-Methylnaphthalene**	10	0.333
Atrazine	10	0.333	♦*Methyl parathion*	10	1.67
Azobenzene	10	0.333	♦♣**2-Methylphenol**	10	0.333
Benzaldehyde	10	1.67	♦♣**3,4-Methylphenol**	10	0.333
Benzidine	100	1.67	♦♣**Naphthalene**	10	0.333
Benzoic acid	50	1.67	♦*1,4-Naphthoquinone*	10	1.67
♦♣**Benzo(a)anthracene**	10	0.333	♦*1-Naphthylamine*	10	0.333
♦♣**Benzo(a)pyrene**	10	0.333	♦*2-Naphthylamine*	10	0.333
♦♣**Benzo(b)fluoranthene**	10	0.333	♦**2-Nitroaniline**	25	0.833
♦**Benzo(g,h,i)perylene**	10	0.333	♦**3-Nitroaniline**	25	0.833
♣*Benzo(j)fluoranthene*	10	0.333	♦**4-Nitroaniline**	25	0.833
♦♣**Benzo(k)fluoranthene**	10	0.333	♦**Nitrobenzene**	10	0.333
♦*Benzyl alcohol*	50	1.67	♦*5-Nitro-o-toluidine*	10	1.67
Biphenyl	10	0.333	♦**2-Nitrophenol**	10	0.333
♦**Bis(2-chloroethoxy) methane**	10	0.333	♦♣**4-Nitrophenol**	25	0.833
♦**Bis(2-chloroethyl) ether**	10	0.333	♦*Nitroquinoline-1-oxide*	10	0.333
♦**Bis(2-chloroisopropyl) ether**	10	0.333	♦*n-Nitrosodiethylamine*	10	0.333
♦♣**Bis(2-ethylhexyl) phthalate**	10	0.333	♦*n-Nitroso-dimethylamine*	10	0.333
♦**4-Bromophenylphenyl ether**	10	0.333	♦*n-Nitrosodi-n-butylamine*	10	1.67
♦♣**Butyl benzyl phthalate**	10	0.333	♦**n-Nitroso-di-n-propylamine**	10	0.333
Caprolactum	10	0.333	♦**n-Nitroso-diphenylamine and Diphenylamine**	10	0.333
Carbazole	10	0.333	♦*n-Nitrosomethylethylamine*	10	0.333
♦**4-Chloro-3-methylphenol**	10	0.333	♦*n-Nitrosomorpholine*	10	1.67
♦**4-Chloroaniline**	10	0.333	♦*n-Nitrosopiperdine*	10	1.67
♦*Chlorobenzilate*	10	1.67	♦*n-Nitrosopyrrolidine*	10	1.67

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Analyte	Water RL µg/L	Soil RL mg/kg	Analyte	Water RL µg/L	Soil RL mg/kg
1-Chloronaphthalene	10	1.67	*Octadecane*	50	0.333
♦**2-Chloronaphthalene**	10	0.333	♦*Parathion*	10	1.67
♦**2-Chlorophenol**	10	0.333	♦*Pentachlorobenzene*	10	1.67
♦**4-Chlorophenylphenyl ether**	10	0.333	♦*Pentachloroethane*	10	0.333
♦♣**Chrysene**	10	0.333	♦*Pentachloronitroben-zene*	10	1.67
♦*cis-Diallate*	10	0.333	♦**Pentachlorophenol**	25	0.833
♦*trans-Diallate*	10	0.333	♦*Phenacetin*	10	1.67
♦**Dibenzofuran**	10	0.333	♦♣**Phenanthrene**	10	0.333
♣*Dibenz(a,h)acridine*	10	0.333	♦♣**Phenol**	10	0.333
Dibenz(a,j)acridine	10	0.333	♦*1,4-Phenylenediamine*	50	0.333
♦♣**Dibenzo(a,h)anthracene**	10	0.333	♦*Phorate*	10	0.333
♦♣**1,2-Dichlorobenzene**	10	0.333	♦*2-Picoline*	10	0.333
♦♣**1,3-Dichlorobenzene**	10	0.333	♦*Pronamide*	10	1.67
♦♣**1,4-Dichlorobenzene**	10	0.333	♦♣**Pyrene**	10	0.333
♦**3,3'-Dichlorobenzidine**	10	0.67	♦♣**Pyridine**	10	0.67
♦**2,4-Dichlorophenol**	10	0.333	♣*Quinoline*	10	0.333
♦*2,6-Dichlorophenol*	20	0.333	♦*Safrole*	10	0.333
3,4-Dichlorophenol	10	1.67	*Terbufos*	50	167
♦♣**Diethyl phthalate**	10	0.333	♦*1,2,4,5-Tetrachloro-benzene*	10	1.67
♦*Dimethoate*	10	1.67	♦*2,3,4,6-Tetrachloro-phenol*	10	0.333
♦*p-Dimethylaminoazobenzene*	10	1.67	♦*Tetraethylpyrophos-phate, Sulfotep*	10	1.67
♦*3,3'-Dimethylbenzidine*	50	0.333	♦*Thionazine*	10	1.67
♦♣*7,12-Dimethylbenz(a)an-thracene*	10	0.333	♣*Thiophenol*	100	3.33
♦*a,a-Dimethylphenethylamine*	50	1.67	♦*o-Toluidine*	10	1.67
♦♣**2,4-Dimethylphenol**	10	0.333	♦**1,2,4-Trichloroben-zene**	10	0.333
♦♣**Dimethyl phthalate**	10	0.333	♦**2,4,5-Trichlorophenol**	25	0.833
♦♣**Di-n-butyl phthalate**	10	0.333	♦**2,4,6-Trichlorophenol**	10	0.333
♦*1,3-Dinitrobenzene*	10	1.67	♦*o,o,o-Triethylphospho-rothioate*	10	1.67
♦**4,6-Dinitro-2-methylphenol**	25	0.833	♦*1,3,5-Trinitrobenzene*	10	0.333
♦♣**2,4-Dinitrophenol**	25	0.833	Acenaphthene, SIM	0.10	0.00333
♦**2,4-Dinitrotoluene**	10	0.333	Acenaphthylene, SIM	0.10	0.00333
♦**2,6-Dinitrotoluene**	10	0.333	Anthracene, SIM	0.10	0.00333
♦♣**Di-n-octyl phthalate**	10	0.333	Benzo(a)anthracene, SIM	0.10	0.00333
♦*Dinoseb*	10	0.333	Benzo(a)pyrene, SIM	0.10	0.00333
1,4-Dioxane	10	0.333	Benzo(b)fluoranthene, SIM	0.10	0.00333
1,2-Diphenylhydrazine	10	0.333	Benzo(g,h,i)perylene, SIM	0.10	0.00333
♦*Disulfoton*	10	1.67	Benzo(k)fluoranthene, SIM	0.10	0.00333
♦*Ethyl methanesulfonate*	10	0.333	Chrysene, SIM	0.10	0.00333
♦*Famphur*	10	0.333	Dibenzo(a,h)anthracene, SIM	0.10	0.00333

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Analyte	Water RL µg/L	Soil RL mg/kg	Analyte	Water RL µg/L	Soil RL mg/kg
♦♣**Fluoranthene**	10	0.333	2,4-Dinitrotoluene, SIM	0.2	0.0067
♦**Fluorene**	10	0.333	2,6-Dinitrotoluene, SIM	0.2	0.0067
♦**Hexachlorobenzene**	10	0.333	Fluoranthene, SIM	0.10	0.00333
♦**Hexachlorobutadiene**	10	0.333	Fluorene, SIM	0.10	0.00333
♦**Hexachlorocyclopentadiene**	10	0.333	Indeno(1,2,3-cd)pyrene, SIM	0.10	0.00333
♦**Hexachloroethane**	10	0.333	1-Methylnaphthalene, SIM	0.10	0.00333
♦*Hexachlorophene*	10	3.33	2-Methylnaphthalene, SIM	0.10	0.00333
♦*Hexachloropropene*	10	1.67	Naphthalene, SIM	0.10	0.00333
♦**Indeno(1,2,3-c,d)pyrene**	10	0.333	Phenanthrene, SIM	0.10	0.00333
♣*Indene*	10	1.67	Pyrene, SIM	0.10	0.00333
♦*Isodrin*	10	0.333	California / Missouri DRO	500	4
♦**Isophorone**	10	0.333	Calilfornia / Missouri ORO	500	4

♦ indicates Appendix IX compound ♣Skinner List compound
Bold compounds are reported in a standard list.
Italicized compounds are only available upon special request by this method.
SIM = Selective Ion Monitoring

1.3 The following compounds may require special treatment when being determined by this method:

- Benzidine may be subject to oxidative losses during solvent concentration, and its chromatographic behavior is poor.
- Hexachlorocyclopentadiene is subject to thermal decomposition in the inlet of the gas chromatograph, chemical reaction in acetone solution, and photochemical decomposition.
- n-Nitrosodimethylamine is difficult to separate from the solvent under the chromatographic conditions described.
- n-Nitrosodiphenylamine decomposes in the gas chromatographic inlet and cannot be separated from diphenylamine.
- Pentachlorophenol, 2,4-dinitrophenol, 4-nitrophenol, benzoic acid, 4,6-dinitro-2-methylphenol, 4-chloro-3-methylphenol, 2-nitroaniline, 3-nitroaniline, 4-chloroaniline, and benzyl alcohol are subject to erratic chromatographic behavior, especially if the GC system is contaminated with high boiling material.
- Pyridine may perform poorly at the GC injection port temperatures listed in the method. Lowering the injection port temperature may reduce the amount of degradation. Use caution if modifying the injection port temperature as the performance of other analytes may be adversely affected.

1.4 The intent of the words "must," "shall," "should," or "will" renders the statement a mandatory requirement of this SOP.

1.5 If for any reason a part of this SOP cannot be followed, seek the guidance of the Department Supervisor or the Laboratory Technical Director. All abnormalities must be noted on the data or the benchsheet and in LIMS. See the corrective action policies in the QA Manual and details in Section 9.0.

2.0 Summary of Method

2.1 The samples are prepared for analysis by gas chromatography/mass spectrometry (GC/MS) using the appropriate sample preparation. See SOPs 3510 / NV03-24 for waters, 3550 / NV03-23 and 3541 / NV03-231 for soils, and 3580 / NV03-106 for oils, and, if necessary, sample

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cleanup procedures.

2.2 The semivolatile compounds are introduced into the GC/MS by injecting the sample extract into a gas chromatograph (GC) with a narrow-bore fused-silica capillary column. The GC column is temperature-programmed to separate the analytes, which are then detected with a mass spectrometer (MS) connected to the gas chromatograph.

2.3 Analytes eluted from the capillary column are introduced into the mass spectrometer via direct connection. Identification of target analytes is accomplished by comparing their mass spectra with the electron impact spectra of standards. Quantitation is accomplished by comparing the response of a major (quantitation) ion relative to an internal standard using at least a multi-point calibration curve.

3.0 Definitions

See the definitions in Appendix 5 of the Quality Assurance Manual.

4.0 Interferences

Contamination by carryover can occur whenever high-concentration and low-concentration samples are sequentially analyzed. To reduce carryover, the sample syringe is rinsed with solvent between sample injections.

5.0 Safety

Employees must abide by the policies and procedures in the Corporate Safety Manual and this document.

This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats, and closed-toe, nonabsorbent shoes are a minimum.

5.1 Specific Safety Concerns or Requirements:

- The gas chromatograph and mass spectrometer contain zones that have elevated temperatures. Be aware of the locations of those zones, and cool them to room temperature prior to working on them.
- The mass spectrometer is under high vacuum. The mass spectrometer must be brought to atmospheric pressure prior to working on the source.
- There are areas of high voltage in both the gas chromatograph and the mass spectrometer. Depending on the type of work involved, either turn the power to the instrument off, or disconnect it from its source of power.

5.2 Primary Materials Used: The following is a list of the materials used in this method, which have a serious or significant hazard rating. **Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

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Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Methanol	Flammable Poison Irritant	200 ppm-TWA	A slight irritant to the mucous membranes. Toxic effects exerted upon nervous system, particularly the optic nerve. Symptoms of overexposure may include headache, drowsiness and dizziness. Methyl alcohol is a defatting agent and may cause skin to become dry and cracked. Skin absorption can occur; symptoms may parallel inhalation exposure. Irritant to the eyes.
Methylene Chloride	Carcinogen Irritant	25 ppm-TWA 125 ppm-STEL	Causes irritation to respiratory tract. Has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting and headache. Causes irritation, redness and pain to the skin and eyes. Prolonged contact can cause burns. Liquid degreases the skin. May be absorbed through skin.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

6.0 Equipment and Supplies

6.1 Instrumentation

- Gas chromatography/mass spectrometer/data system
  - Gas chromatograph (HP or Agilent) -- Analytical system complete with a temperature-programmable gas chromatograph suitable for split/splitless injection and all required accessories, including syringes, analytical columns, and gases. The capillary column is directly coupled to the source.
  - Column -- 30 m x 0.25 mm ID with a 0.25 µm film thickness silicone-coated fused-silica capillary column (Phenomonex ZB-5 or equivalent).
  - Mass spectrometer capable of scanning from 35 to 500 amu every 1 second less, using 70 volts (nominal) electron energy in the electron impact ionization mode. The mass spectrometer is capable of producing a mass spectrum for decafluorotriphenylphosphine (DFTPP) which meets the criteria in Table 2 when 1µL of the GC/MS tuning standard is injected (50 ng or less of DFTPP).
  - Data system (Chemstation with Enviroquant) -- A computer system is interfaced to the mass spectrometer. The system allows the continuous acquisition and storage on machine-readable media of all mass spectra obtained throughout the duration of the chromatographic program. The computer has software that can search any GC/MS data file for ions of a specific mass and that can plot such ion abundances versus time or scan number. This type of plot is defined as an Extracted Ion Current Profile (EICP). Software is also available that allows integrating the abundances in any EICP between specified time or scan-number limits. The EPA/NIST Mass Spectral Library is also available.
  - Suggested operating conditions (may vary by instrument; see maintenance log for current program):

Mass range:	35-500 amu
Scan time:	1 second/scan
Initial temperature:	40°C hold for 2 minutes
Temperature program:	Rate 1: 15°C/minute to 160°C
	Rate 2: 10°C/minute to 320°C
Final temperature:	320°C hold for at least 1.5 minute.
Injector temperature:	240-250°C
Transfer line temperature:	300°C

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Source temperature:	According to manufacturer's specifications (nominally 250 – 275°C)
Injector:	Grob-type, split-less
Injection volume:	1 µL
Carrier gas:	Helium at 1 mL/minute

6.2 Supplies

- Microsyringe – 10-25 µL.
- Balance -- Analytical, capable of weighing 0.0001 g
- Glass vials -- glass with PTFE (polytetrafluoroethylene)-lined screw-caps or crimp tops.
- Volumetric flasks, Class A – appropriate sizes with ground-glass stoppers.

7.0 Reagents and Standards

7.1 Reagent grade chemicals are used in all tests. Unless otherwise, indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

7.2 Reagent water, analyte-free, ASTM Type II or equivalent.

7.3 Stock Calibration Standards: Commercially prepared, certified stock standards are purchased:

- The primary standard for the typical 8270 compound list is from Ultra Scientific CUS-5160, or equivalent, with the required targets at 200 µg/mL.
- For PAHs by SIM, use Accustandard Z-014G-FL, or equivalent, with the target PAHs at 2000 µg/mL.
- For Appendix IX and miscellaneous compounds, primary source standards are purchased from NSI; equivalent substitutes are acceptable.

Analyte/Analyte Group	NSI Catalog Number	Concentration (µg/mL)
AIX Mix	C-426-11	2000
Acid Extractables II	C415-14	2000
Amines	C-412	2000
Aramite	922-05-02	2000
a,a-Dimethylphenylamine	922-05-02	2000
Benzidines	C-411	2000
BNA II mix	C-413-10	2000
B/N III mix	C-414-13	2000
Hexachlorophene	323-03-04	5000
Sulfonates	C-416-08	2000
8270 OP Pest	C-417-13	2000

7.4 Matrix Spike and Laboratory Control Standard contains all targets to be reported on the samples. The same compounds mentioned in Section 7.3 are designated as the SPCCs and CCCs for 8270C.

- For both a long semivolatile list and the PAH list by SIM, purchase as the second source a 100 µg/mL standard, NSI Catalog # c-408-50x, or equivalent.
- For Appendix IX and miscellaneous compounds, these second source standards are acceptable, as well as equivalents:

Analyte/Analyte Group	RestekCatalog Number	Concentration (µg/mL)

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AIX #1 Mix	31625	2000
AIX #2 Mix	31806	2000
Calibration Mix	31618	2000
OP Mix	32419	2000

7.5 Internal standard solutions: The internal standards are 1,4-dichlorobenzene-d_4, naphthalene-d_8, acenaphthene-d_{10}, phenanthrene-d_{10}, chrysene-d_{12}, and perylene-d_{12}.

- Purchase certified, internal standard at 4000 µg/mL, NSI C-394, or equivalent.

7.6 GC/MS Tuning Standard: A methylene chloride solution containing 50 µg/mL of decafluorotriphenylphosphine (DFTPP) is prepared. The standard also contains 50 µg/mL each of 4, 4'-DDT, pentachlorophenol, and benzidine to verify injection port inertness and GC column performance.

- Purchase the tuning standard at 1000 µg/mL from Ultra Scientific, Catalog GCM-150, or equivalent.

7.7 Surrogate standards: The surrogates are phenol-d_5, 2-fluorophenol, 2,4,6-tribromophenol, nitrobenzene-d_5, 2-fluorobiphenyl, and p-terphenyl-d_{14}.

- Purchase the acid/base/neutral and PAH SIM surrogates from NSI, CUS-7070, or equivalent, at 50 µg/mL.

7.8 Acetone, hexane, methylene chloride, isooctane, carbon disulfide, toluene, and other appropriate solvents, commercial source.

7.9 Sodium sulfate for blank and LCS soil matrix.

7.10 Transfer the stock standard solutions into bottles with PTFE-lined screw-caps. Store, protected from light, at -10°C or less or as recommended by the standard manufacturer. Stock standard solutions must be checked frequently for signs of degradation or evaporation, especially just prior to preparing calibration standards from them. Replace after **one year or sooner** if comparison with quality control check samples indicates a problem, or if the vendor specifies an expiration date sooner than one year.

8.0 Sample Collection, Preservation, Shipment and Storage

Matrix	Sample Container	Min. Sample Size	Preservation	Holding Time	Reference
Water	3 L, amber glass with Teflon®-lined cap	1 L	Cool 0-6°C. Keep in dark.	7 days from collection until extraction, 40 days after extraction	SW-846 Chapter 2
Soil	4 oz. glass jar with Teflon®-lined cap	30 g	Cool 0-6°C.	14 days from collection until extraction, 40 days after extraction	

9.0 Quality Control

The laboratory maintains a formal quality assurance program and records to document the quality of the data generated.

Certain quality control and reporting criteria may vary depending on whether SW-846 8000B or 8000C criteria are required. In these cases, both sets of criteria have been noted in this SOP. 8000C criteria are required to be applied ONLY to Arizona and Washington samples. All other samples must be processed against referenced 8000B criteria. Exceptions may be required on a project-specific basis.

9.1 Sample QC:

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The following QC samples are run with **each batch of no more than 20 samples, except for samples from OK and WY, which have a maximum of 10 samples per batch.**			
QC Check	**Frequency**	**Acceptance Criteria[1]**	**Corrective Action[2]**
Method blank	One per analytical prep batch	No analytes detected ≥ ½ RL or MDL, whichever is greater	Correct problem then re-prep[3] and analyze method blank and all samples processed with the contaminated blank.
LCS[6] for all analytes (2nd source)[6]	One[6] per prep batch	See Control Limits Manual and footnote 4 below	Correct problem then re-prep[4] and analyze the LCS and all samples in the affected analytical batch.[4] If high and target is ND, OK to report.
MS/MSD (2nd source)	One per batch per matrix, if insufficient sample for MS/MSD, qualify data[3]	See Control Limits Manual	None (the LCS is used to evaluate to determine if the batch is acceptable).
Surrogate(s)	Every sample, spike, standard, and blank	See Control Limits Manual[5]	Check system, re-analyze, re-prep[3, 5].

[1]This is a summary of the acceptance criteria.
[2]All abnormalities must be noted on the data, the benchsheet and in LIMS.
[3]If unable to re-prep samples because of insufficient sample volume or the holding time has expired, then place a comment on the benchsheet and in LIMS.
[4]If the LCS exceeds the upper control limit AND a sample from that batch is greater than the RL, re-prep and re-analyze the batch. If the LCS exceeds the upper control limit AND the samples from that batch is less than the RL, the data is acceptable to report.
[5]If the surrogate % recovery exceeds the upper control limit AND a sample result is positive above the RL, re-prep and re-analyze the batch. If the surrogate % recovery exceeds the upper control limit AND the sample is less than the RL, data is acceptable to report. If the surrogate % recovery is lower than the lower control limit, re-prep the sample. OH VAP requires all surrogates to be in control; otherwise, the samples must be re-prepared and re-analyzed.
[6]LCSD is required for AZ, MA, TX, WV.

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- A **Method blank** is extracted with every batch of samples. Evaluate the blank to be less than one-half the RL or the MDL, whichever is greater. If any analyte is detected at a level exceeding the criterion, re-analyze the blank. If the contamination is persistent, all samples in the batch must be re-extracted unless the target is 10 times the blank. (OH VAP does not allow this; therefore, re-prep OH VAP samples.). If insufficient sample remains for re-extraction or holding time is already exceeded, then the "B" flag is used on the final reports to identify positive target compounds that were also positive in the method blank.
- A **Laboratory Control Sample** (LCS) is included with each analytical batch. The LCS consists of an aliquot of a clean (control) matrix similar to the sample matrix and of the same weight or volume (reagent water for water batches, sodium sulfate for soil batches). It is spiked with the same analytes at the same concentrations as the matrix spike. When the results of the matrix spike analysis indicate a potential problem due to the sample matrix itself, the LCS results are used to verify that the laboratory can perform the analysis in a clean matrix. **All analytes must meet the LCS QC criteria** (laboratory historical limits in the Control Limits Manual), **or the samples must be re-extracted if sufficient volume of sample remains.** However, if the LCS is high, and a target is ND, it is acceptable to report the result.
  - The LCS spike is from a **different source than the calibration standards**. Using the 100 µg/mL LCS/MS/MSD standard:
    - **For Non-SIM batches:**
      - Water: add 500 µL of the standard per liter reagent water before extraction by Method 3510C.
      - Soil: add 500 µL of the standard per 30 gram sodium sulfate before extraction.

- TCLP: add 1 mL of the standard/500 mL TCLP extraction fluid before extraction by Method 3510C.
- The final concentration is 50 µg/mL in the above extracts.
- **For SIM batches:**
  - Water: add 1 mL of a 100 X dilution of the NSI standard per liter reagent water.
  - Soil: add 1 mL of a 100 X dilution of the NSI standard per 30 g sodium sulfate.
  - The final concentration in the extracts is 1.0 µg/mL.

- **Matrix Spike / Matrix Spike Duplicate**: Documenting the effect of the matrix includes the analysis of at least one matrix spike/matrix spike duplicate pair. The percent recovery and RPD are compared to the acceptance criteria (laboratory historical limits in Control Limits Manual).
  - The MS/MSD spike is from a **different source** than the calibration standards. Using the 100 µg/mL LCS/MS/MSD standard:
    - **For Non-SIM batches**:
      - Water: add 500 µL of the standard per liter client sample.
      - Soil: add 500 µL of the standard to 30 g client sample.
      - TCLP: add 1 mL of the standard per 500 mL client TCLP extract.
      - The final concentration is 50.0 µg/mL in the above extracts.
    - **For SIM batches**:
      - Water: add 1 mL of a 100 X dilution of the NSI standard per liter reagent water.
      - Soil: add 1 mL of 100 X dilution per 30 g client sample.
      - The final concentration in the extracts is 1.0 µg/mL.
- **Surrogate recoveries** - The laboratory evaluates surrogate recovery data from individual samples versus the surrogate control limits developed by the laboratory. The limits for surrogate recoveries are updated biannually (see TestAmerica Nashville's current Control Limits Manual (CLM)). If any surrogate is outside QC limits, and there is no obvious matrix interference, then re-analyze and/or re-extract the sample. If surrogates are still outside limits, flag the data in LIMS. However, if high and all results are non-detect, results are reportable. If surrogate recoveries are low, re-prep the batch.
  - **For Non-SIM**, add 1000 µL of the surrogate standard at a concentration of 50 µg/mL to each sample and batch QC samples prior to extraction for a 50 µg/mL concentration in the extract.
  - **For SIM,** prepare a 1 µg/mL standard (500 µL surrogate standard) to 500 mL in methanol. Add 1.0 mL to samples and QC (blanks, MS/MSD and LCS) prior to extraction. The concentration is 1.0 µg/mL in the extract.

9.2 Instrument QC

QC Check	Frequency	Acceptance Criteria[2]	Corrective Action[3]
GC/MS Tuning			
a. Check of mass spectral ion intensities[1], i.e., Tune	Prior to initial calibration or Continuing calibration verification, every 12 hours.	See below in this section for GC/MS Tuning criteria.	Retune the instrument and verify (instrument maintenance may be needed).
b. Column Breakdown	Prior to initial calibration or Continuing calibration verification, every 12 hours.	Breakdown ratio ≤ 20%.	Injector or column maintenance and re-calibration.
c. Tailing Factor	Prior to initial calibration or Continuing calibration verification, every 12 hours.	8270C / 8270D Benzidine 3 / 2 Pentachlorophenol 5 / 2	Injector or column maintenance and re-calibration.

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Minimum five-point initial calibration for all target analytes	Initial calibration prior to sample analysis. Perform instrument re-calibration once per year minimum.	8270C: SPCCs average RF ≥ 0.050 and %RSD for RFs for CCCs ≤ 30% and all other target analytes %RSD for RF ≤ 15% If %RSD is > 15%, linear regression $r^2 \geq 0.99$, $r \geq 0.995$. 8270D: The minimum RF for all compounds in Attachment 4 must be met. All targets RSD ≤ 20% or use linear regression. If %RSD is > 15%, linear regression $r^2 \geq 0.99$, $r \geq 0.995$.	Correct problem then repeat initial calibration.
Initial calibration verification (ICV) must be from a 2nd source.	Immediately following five-point initial calibration.	All analytes within 30% of expected value.	Correct problem then repeat initial calibration.
Initial calibration blank	Immediately after ICV	All analytes < MDL	Correct problem, re-calibrate.
Continuing calibration verification (CCV)	Daily, before sample analysis and every 12 hours of analysis time.	8270C: SPCCs average RF ≥ 0.050 and CCCs: ≤20% difference (when using RFs) or drift (when using least squares regression). 8270D: The minimum RF for all compounds listed in Attachment 4 must be met and the percent difference for each target compound ≤ 20%.	Correct problem then repeat initial calibration and re-analyze all samples since last successful CCV.
Internal Standards	Every sample/standard and blank.	Retention time ±30 seconds from retention time of the mid-point std. in the ICAL for CCV. EICP area within -50% to +100% of ICAL mid-point std for the CCV and –50% to +100% of the prior CCV for the samples. See footnote 4 below.	Inspect mass spectrometer and GC for malfunctions; mandatory re-analysis of samples analyzed while system was malfunctioning (dilution of the sample may be required, see the supervisor or the technical director for advice).
Relative Retention Time Window	Each sample.	Relative retention time (RRT) of the analyte within 0.06 RRT units of the RRT of the internal standard.	Correct problem then reprocess or re-analyze all samples analyzed since the last retention time check.
MDL verification (extracted)	Minimum yearly.	Detectible	Re-evaluate MDL standard used and MDL; see Technical Director.

[1]8270 requires DFTPP.
[2]This is a summary of the acceptance criteria.
[3]All abnormalities must be noted on the data, the benchsheet and in LIMS.
[4]Target compounds associated with failed internal standards must be re-analyzed (undiluted if possible) if additional sample is available; if not available, qualify data in LIMS.

- **GC/MS Tuning**
  - Prior to the analysis of samples or calibration standards, the GC/MS system is hardware-tuned using a 50 ng or less injection of DFTPP (in the GC/MS Tuning Standard).
  - The 50 µg/mL standard is prepared by adding 2.8 mL of 1000 µg/mL stock standard to 56 mL methylene chloride.
  - Analyses **must** not begin until the tuning criteria are met, and these criteria must be demonstrated at the beginning of each 12-hour shift. Three options are available for acquiring the spectra for reference to meet the DFTPP tuning requirements:

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Option 1 It is recommended that each initial tune verification utilize the "Autofind" function and be set up to look at the apex ±1 scan and average the three scans. Background correction is required prior to the start of the peak but no more than 20 scans before. Background correction **cannot** include any part of the target peak. Sometimes the instrument does not always correctly identify the apex on some peaks when the peak is not perfectly shaped. It is acceptable to manually identify and average the apex peak ± 1 scan and background correct

Option 2 The entire peak may be averaged and background-corrected. Average scans from 0.1 minute before to 0.1 minute after peak.

Option 3 A single scan at the apex (only) may also be used for the evaluation of the tune. Background correction is required.

Note: It is acceptable to adjust parameters within the specifications set by the manufacturer or the analytical method to properly tune the instrument. If the tune verification does not pass, it may be necessary to clean the source or perform additional maintenance. Document any maintenance in the instrument log. **Excessive adjusting (more than two tries) without clear documentation is not allowed. No more than two consecutive tunes may be attempted. Perform necessary maintenance.**

- All subsequent standards, samples, controls, and blanks associated with a DFTPP tune must use the identical mass spectrometer instrument conditions.
- Use the DFTPP mass intensity criteria as follows as tuning acceptance criteria.

DFTPP Key Ions and Ion Abundance Criteria

Mass	m/z Abundance criteria
51	30-60 percent of mass 198.
68	Less than 2 percent of mass 69.
70	Less than 2 percent of mass 69.
127	40-60 percent of mass 198.
197	Less than 1 percent of mass 198.
198	Base peak, 100 percent relative abundance.
199	5-9 percent of mass 198.
275	10-30 percent of mass 198.
365	Greater than 1 percent of mass 198.
441	Present but less than mass 443.
442	Greater than 40 percent of mass 198.
443	17-23 percent of mass 442.

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- **Breakdown Standard**: The GC/MS Tuning Standard is also used to assess the injection port inertness by evaluating the degradation of DDT to DDE and DDD. This ratio must **not** exceed 20%; see Section 11 for **percent breakdown** calculation. Perform injector or column maintenance and recalibrate if the ratio maximum is exceeded for either compound. The breakdown of DDT is measured **before** verification standards and samples are analyzed and every 12 hours throughout the sequence.
- **Tailing Factor**: To evaluate the GC column, benzidine and pentachlorophenol (in the GC/MS Tuning Standard) must be present at their normal responses and evaluated for peak tailing. The benzidine and pentachlorophenol tailing factors are calculated by the following equation:

Tailing factor = BC/AB

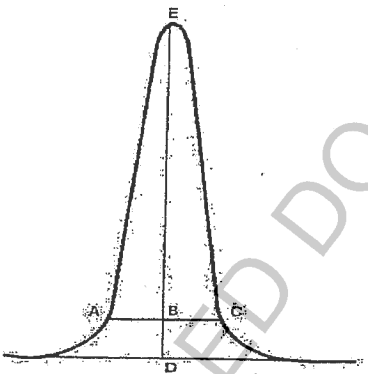
Maximum Tailing Factor Ratios

Tailing Factor Compounds	8270C	8270D

Benzidine	3	2
Pentachlorophenol	5	2

where the peak is defined as follows: AC is the width at 10% height. DE is the height of peak and B is the height at 10% DE. This equation compares the width of the back half of the peak to the width of the front half of the peak at 10% of the height. (See Figure 1 for an example tailing factor calculation.)

FIGURE 1
TAILING FACTOR CALCULATION



$$\text{TAILING FACTOR} = \frac{BC}{AB}$$

Example calculation: Peak Height = DE = 100 mm
10% Peak Height = BD = 10 mm
Peak Width at 10% Peak Height = AC = 23 mm
AB = 11 mm
BC = 12 mm

$$\text{Therefore: Tailing Factor} = \frac{12}{11} = 1.1$$

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If all of the specified criteria are met, generate a hardcopy of the spectrum, the mass abundance data and the parameters under which the scans were acquired. This data is filed in the batch for documentation.

- **Calibration Standards**: See Section 10.2.
- **Initial Calibration System Performance Check Compounds (SPCCs):** A system performance check is performed to ensure that minimum average RFs are met before the calibration curve is used.
  - **For 8270C**: The SPCCs are

System Performance Check Standards (SPCCs)	
Base/Neutral Fraction	**Acid Fraction**
n-Nitrosodi-n-propylamine	2,4-Dinitrophenol
Hexachlorocyclopentadiene	4-Nitrophenol

The **minimum acceptable average RF for the SPCCs is 0.050.** They typically have very low RFs (0.1-0.2) and tend to decrease in response as the chromatographic system begins to deteriorate or the standard material begins to deteriorate. They are usually the first to show poor performance. Therefore, they must meet the minimum requirement when the system is calibrated.

- **For 8270D**, see Attachment 4 for required minimum response factor criteria for target analytes.
- If the minimum response factors are not met, the system must be evaluated, and corrective action is taken before sample analysis begins. Possible problems include standard mixture degradation, injection port inlet contamination, contamination at the front end of the analytical column, and active sites in the column or chromatographic system. **This check must be met before sample analysis begins.**

- **Initial Calibration Calibration Check Compounds (CCCs)**: The purpose of the CCCs is to evaluate the calibration from the standpoint of the integrity of the system. High variability for these compounds may be indicative of system leaks or reactive sites on the column. Meeting the CCC criteria is not a substitute for successful calibration of the target analytes. The CCCs are:

Calibration Check Compounds (CCC)	
Base/Neutral Fraction	**Acid Fraction**
Acenaphthene*	4-Chloro-3-methylphenol
1,4-Dichlorobenzene	2,4-Dichlorophenol
Hexachlorobutadiene	2-Nitrophenol
Diphenylamine	Phenol
Di-n-octyl phthalate	Pentachlorophenol
Fluoranthene*	2,4,6-Trichlorophenol
Benzo(a)pyrene*	

*For PAH SIM standard

- Calculate the mean response factor and the relative standard deviation (RSD) of the response factors for each target analyte.

Initial CCCs RSD Differences	
8270C	**8270D**
The RSD must be less than or equal to **15%** for each target analyte; if not, see the section on linearity of target analytes in Section 10.2. However, the RSD for each individual CCC must be less than or equal to 30%. If the RSD of any CCC is greater than 30%, then the chromatographic system is too reactive for analysis to begin. Clean or replace the injector liner and/or capillary column, then repeat the calibration procedure.	The RSD must be less than or equal to **20%** for each target analyte; if not, see the section on linearity of target analytes in Section 10.2. If not, check errors in standard preparation, the possible presence of active sites in the GC system, poor chromatographic behaviors for analytes.

- The **Initial Calibration Verification (ICV)** is a second-source standard run immediately after the initial calibration. The acceptance limits are **70-130%** recovery.
  - Add 250 µL of the second-source standard to 250 µL methylene chloride in an amber vial to prepare an ICV standard at 50 µg/mL.
  - For PAHs by SIM, use the second-source standard with the target PAHs at 2000 µg/mL. A 10 µg/mL intermediate is made by taking 50 µL of the stock standard along with 20 µL of the base/neutral surrogates. The ICV at 1 µg/mL is made by taking 50 µL of

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intermediate into 450 µL of methylene chloride in an amber vial.
- If ICV acceptance criterion is not met, correct the problem and re-calibrate.

- **Initial Calibration Blank**: a reagent/solvent blank analyzed after the ICV to ensure the system is free of contaminants (< MDL). If not contaminant-free, re-run and/or perform system maintenance.
- The **Continuing Calibration Verification standard (CCV)** is evaluated each day (or every 12 hours) that analysis is performed to determine if the chromatographic system is operating properly.
  - Prepare a daily CCV at 50 µg/mL by adding 100 µL of the primary stock solution to 300 µL methylene chloride in an amber vial.
  - For PAHs by SIM, use the primary stock standard with the target PAHs at 2000 µg/mL. A 10 µg/mL intermediate is made by taking 50 µL of the stock standard along with 20 µL of the base/neutral surrogates. A daily CCV at 1 µg/mL is made by taking 50 µL of intermediate into 450 µL of methylene chloride in an amber vial.
  - The calibration verification standard is prepared at least weekly and stored at 4°C or less.
  - Each **SPCC** in the calibration verification (CCV) standard must meet a **minimum response factor of 0.050**. This is the same check and same corrective action that are applied during the initial calibration. **For 8270D**, see Attachment 4 for required minimum response factor criteria for target analytes.
  - After the system performance check is met, the **CCCs** are used to check the ongoing validity of the initial calibration. Use percent difference when performing the average response factor model calibration. Use percent drift when calibrating using a regression fit model. If the bias is high and targets are non-detected, the data may be reported.

CCC % Difference Evaluation Criteria	
8270C	**8270D**
CCCs and all other target compounds require an RF ≤ 20%; however, up to 5 non-CCC target compounds may be ≤ 40%.	If the percent difference for each target compound is less than or equal to **20%**, then the initial calibration is assumed to be valid. If the criterion is not met (i. e., greater than 20% difference or drift) for any target, then corrective action is taken prior to the analysis of samples.

  - If the CCV criteria cannot be met, a new initial calibration must be generated.
- **Continuing Calibration Blank (CCB):** The CCB is run after each CCV. If the result is not ≤ MDL or ½ RL, correct the problem and re-run.
- **Internal standards** are added to every sample, standard, and QA/QC.
  - **Retention time** - The retention times of the internal standards in the continuing calibration verification (CCV) standard must be evaluated immediately after or during data acquisition. If the retention time for any internal standard changes by more than **30 seconds** from that in the mid-point standard level of the **most recent initial calibration** sequence, then the chromatographic system must be inspected for malfunctions and corrections must be made. When corrections are made, reanalysis of samples analyzed while the system was malfunctioning is required.
  - **Response** - If the EICP area for any of the internal standards in the continuing calibration verification (CCV) standard changes by a factor of two (-50% to +100%) from that in the mid-point standard level of the **most recent initial calibration sequence**, the mass spectrometer must be inspected for malfunctions and corrections must be made. When corrections are made, reanalysis of samples analyzed while the system was malfunctioning is required.

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- The laboratory re-analyzes any sample where the internal standard fails and there is no evidence of matrix interference. If there is no matrix interference, the sample must be reanalyzed at the original dilution.
  - If the internal standard is within criteria, report the second analysis.
  - If the internal standard is still outside of criteria, the sample must be analyzed at a second dilution.
  - If the internal standard still does not meet criteria, the sample must be diluted until the internal standard meets criteria. Multiple runs may be required.
- The target analytes are quantitated with specific internal standards as shown in this table:

Semivolatile Internal Standards with Corresponding Analytes Assigned for Quantitation

1,4-Dichlorobenzene-d_4	**Naphthalene-d_8**	**Acenaphthene-d_{10}**
Aniline	Benzoic acid	Acenaphthene
Benzyl alcohol	Bis(2-chloroethoxy) methane	Acenaphthylene
Bis(2-chloroethyl) ether	4-Chloroaniline	2-Chloronaphthalene
Bis(2-chloroisopropyl) ether	4-Chloro-3-methylphenol	4-Chlorophenyl phenyl ether
2-Chlorophenol	2,4-Dichlorophenol	Dibenzofuran
1,3-Dichlorobenzene	2,4-Dimethylphenol	Diethyl phthalate
1,4-Dichlorobenzene	Hexachlorobutadiene	Dimethyl phthalate
1,2-Dichlorobenzene	Isophorone	2,4-Dinitrophenol
2-Fluorophenol (surr)	2-Methylnaphthalene	2,4-Dinitrotoluene
Hexachloroethane	Naphthalene	2,6-Dinitrotoluene
2-Methylphenol	Nitrobenzene	Fluorene
3,4-Methylphenol	Nitrobenzene-d_8 (surr)	2-Fluorobiphenyl (surr)
n-Nitrosodimethylamine	2-Nitrophenol	Hexachlorocyclopentadiene
n-Nitroso-di-n-propyl- amine	1,2,4-Trichlorobenzene	2-Nitroaniline
Phenol	1-Methylnapthalene	3-Nitroaniline
Phenol-d_5 (surr)	Hexachloropropene	4-Nitroaniline
Pyridine	2,6-Dichlorophenol	4-Nitrophenol
2-Chlorophenol-d_4 (surr)	n-Nitrosodi-n-butylamine	2,4,6-Trichlorophenol
1,2-Dichlorobenzene-d_4 (surr)	1,4-Phenylenediamine	2,4,5-Trichlorophenol
1,4-Dioxane	trans-Isosafrole	1,2-Diphenylhydrazine
Pyridine	1,2,4,5-Tetrachlorobenzene	1,3-Dinitrobenzene
2-Picoline	cis-Isosafrole	Pentachlorobenzene
N-Nitrosomethylethylamine	Safrole	1-Naphthaleneamine
Methyl-methoanesulfonate	1-Chloronaphthalene	2-Naphthaleneamine
n-Nitrosodiethylamine	1,4-Naphthoquinone	2,3,4,6-Tetrachlorophenol
Ethylmethanesulfonate		Diphenylamine
Pentachloroethane		5-Nitro-o-Toluidine
Acetonphenone		trans-Diallate
n-Nitrosopyrrolidine		cis-Diallate
2-Toluidine		1,3,5-Trinitrobenzene
n-Nitrosomorpholine		Phenacetin
n-Nitrosopiperidine		4-Aminobiphenyl
Phenanthrene-d_{10}	**Chrysene-d_{12}**	**Perylene-d_{12}**
Anthracene	Benzidine	Benzo(b)fluoranthene
4-Bromophenyl phenyl ether	Benzo(a)anthracene	Benzo(k)fluoranthene
Di-n-butyl phthalate	Bis(2-ethylhexyl) phthalate	Benzo(g,h,i)perylene
4,6-Dinitro-2-methylphenol	Butyl benzyl phthalate	Benzo(a)pyrene
Diphenylamine	Chrysene	Dibenz(a, h)anthracene
Fluoranthene	3,3'-Dichlorobenzidine	Di-n-octyl phthalate
Hexachlorobenzene	Pyrene	Indeno(1,2,3-cd)pyrene

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n-Nirosodiphenylamine	Terphenyl-d_{14} (surr)	Dibenz(a,j)acridine
Pentachlorophenol	4,4'Methylenebis(2-chloro-aniline)	7,12-Dimethylbenz(a)anthracene
Phenanthrene	3,3'-Dichlorobenzidine	
Carbazole	Aramite	
Bis (2-ethylhexyl)adipate	3-Methylcholanthrene	
Tribromophenol (surr)		
Thionazin		
Pronamide		
Pentachloronitrobenzene		
Dinoseb		
Sulfotepp		
Phorate		
Dimethoate		
Disulfoton		
4-Nitroquinoline-N-oxide		
Methapyrilene		
Isodrin		
Methyl Parathion		
Benzidine		
Parathion		
Hexachlorophene		
Kepone		
4-Dimethylaminozobenzene		
Chlorobenzilate		
3,3'-Dimethylbenzidine		
2-Acetylaminofluorene		

(surr)= surrogate

- The internal standards selected permit most of the components of interest in a chromatogram to have retention times of 0.80-1.20 relative to one of the internal standards. Use the base peak ion from the specific internal standard as the primary ion for quantitation. If interferences are noted, use the next most intense ion as the quantitation ion (i. e., for 1, 4-dichlorobenzene-d_4, use 152 m/z for quantitation).
- Dilute the 4000 µg/mL internal standard by 2x with methylene chloride. The resulting solution contains each internal standard mixture at a concentration of 2000 µg/mL. Each 0.5 mL sample extract undergoing analysis is spiked with 10 µL of the internal standard solution, resulting in a concentration of 40 µg/mL of each internal standard.
- For SIM, dilute the 2000 µg/mL internal standard mix by 10x with methylene chloride for a 200 µg/mL standard. Each 0.5 mL of sample extract undergoing analysis is spiked with 10 µL of internal standard solution, resulting in a concentration of 4 µg/mL of each internal standard.

- **Evaluation of target analyte retention time:** The relative retention time (RRT) of each target analyte in each calibration standard must agree within 0.06 RRT units. Late-eluting target analytes usually have much better agreement. This criterion is met with the use of a ± 0.25 minute retention time window. Representative retention times are shown in Attachments 1 and 2.
- **Method Detection Limit Verification (MDLV):** Annually, verify that the MDL is detectible; if not, re-evaluate the MDL.

10.0 Procedure

10.1 Sample Preparation

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Matrix	Sample Size
Water	1000 mL
Soil	30 grams

- Samples are nominally prepared by one of the following methods prior to GC/MS analysis:

Matrix	Methods	SOP #
Water	3510	NV03-24
Soil/sediment	3541, 3550	NV03-231, NV03-25
Oily Waste	3580	NV03-106

- QC samples and client samples must be extracted by the same preparation method.
- All calibration standards, QC samples, and client samples are introduced into the GC/MS using the same injection volume, IS and SS concentrations, and instrument conditions.

10.2 Calibration and Daily Continuing Calibration Verification

- Initially and/or daily, evaluate the DFTPP tune criteria (Section 9.2).
- Evaluate the percent breakdown of DDT (Section 9.2).
- Evaluate the tailing factors for benzidine and pentachlorophenol (Section 9.2).
- See Section 11 for equations.

Initial calibration

1 Prepare calibration standards at five (minimum) different concentrations.

Concentration (µg/mL)	µL 200 µg/mL standard/500 µL
2	5
10	25
20	50
50	125
80	200
100	250

- At least one of the calibration standards corresponds to a sample concentration **at or below the laboratory reporting limit (RL)**. The remaining standards correspond to the working range of the GC/MS system.
- Each standard contains each analyte to be reported. These target analytes may not include the entire list of analytes for which the method has been demonstrated; however, the laboratory **must not** report a quantitative result for a target analyte that was not included in the calibration standard(s).
- Surrogates are included at the same concentrations.
- The internal standards are at a constant 40 µg/mL. Each 0.5 mL aliquot of calibration standard is spiked with 10 µL of the internal standard solution prior to analysis.

2 For SIM, calibration standards are diluted from the intermediate standard solution to give the following concentrations:

Concentration (µg/mL)	µL 200 µg/mL standard/500 µL
0.05	2.5
0.1	5

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	<table><tr><td>0.5</td><td>25</td></tr><tr><td>1</td><td>50</td></tr><tr><td>5</td><td>250</td></tr><tr><td>10</td><td>500</td></tr></table> • Surrogates are included at the same concentrations. • The internal standards are at a constant 4 µg/mL. • See Attachments 2 and 3 regarding SIM Mass groups.
3	Analyze 1 µL of each calibration standard (containing internal standards) and tabulate the area of the primary characteristic ion against concentration for each target analyte. See Attachment 1. **Two characteristic ions must be valid for the low standard to be used.**
4	Calculate response factors (RFs) for each target analyte relative to one of the internal standards.
5	Evaluate the **system performance check compounds (SPCCs):** The minimum acceptable average RF for these compounds is 0.050 for 8270C. For 8270D, see Attachment 4. This check must be met before sample analysis begins.
6	Evaluate the **calibration check compounds (CCCs):** If the RSD of any CCC is greater than 8270C or 8270D criteria, then correct the chromatographic system reactivity before analysis begins.
7	Evaluate the **minimum response factors.**
8	Evaluate the **retention times.**
9	Evaluate the **linearity of target analytes** - If the RSD of any target analytes is 15% or less, then the relative response factor is assumed to be constant over the calibration range, and the average relative response factor is used for quantitation. If the RSD of any target analyte is greater than 15% (8270D 20%), linear regression is used for calibration. The correlation coefficient r^2 must be at least 0.99 ($r \leq 0.995$). If the calibration is not considered linear by either %RSD or linear regression, then correct the problem and re-calibrate. See Section 11 for equations and information on linear regression calibration. **For 8270D only:** The method of linear regression analysis has the potential for a significant bias to the lower portion of a calibration curve. When calculating the calibration curves using the linear regression model, a minimum quantitation check on the viability of the lower calibration point must be performed by re-fitting the response from the low concentration calibration standard back into the curve. It is not necessary to re-analyze a low concentration standard; rather the data system can recalculate the concentrations as if it were an unknown sample. The recalculated concentration of the low calibration point should be within ± 30% of the standard's true concentration. Analytes which do not meet the minimum quantitation calibration re-fitting criteria are considered "out of control" and corrective action such as redefining the lower limit of quantitation and/or reporting those "out of control" target analytes as estimated when the concentration is at or near the lowest calibration point may be appropriate.
11	Evaluate the success of the initial calibration by running an Initial Calibration Verification (ICV).
12	Evaluate the Initial Calibration Blank to be sure it is free of contaminants.

Initial Calibration Sequence Summary

1	DFTPP Tuning Criteria
2	DDT Breakdown Percentage
3	Benzidine and Pentachlorophenol Tailing Factors
4	Calibration Standards

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5	ICV
6	ICB

Daily continuing calibration verification - Calibration verification is performed at the beginning of **each** 12-hour analytical shift.	
1	The initial calibration for each compound of interest is verified once every 12 hours and prior to sample analysis by analyzing a continuing calibration verification (CCV) standard.
2	Evaluate the **system performance check compounds (SPCCs):** Each SPCC in the calibration verification (CCV) standard must meet the **minimum response factor criteria** for 8270C or 8270D.
3	Evaluate the **calibration check compounds (CCCs)** for 8270C or 8270D criteria:
4	Evaluate the **minimum response factors** of each of the most common target analytes in the calibration verification standard.
5	Evaluate the **internal standard retention times** in the CCV.
6	Evaluate the **internal standard responses**.
7	Analyze an extraction blank after the continuing calibration standard, or at any other time during the analytical shift, to ensure that the total system (introduction device, transfer lines and GC/MS system) is free of contaminants.

10.3 Sample Analysis

1	Allow the sample extract to warm to room temperature. Just prior to analysis, add 10 µL of the internal standard solution to the 0.5 mL concentrated sample extract.
2	Inject a 1 µL aliquot of the sample extract into the GC/MS system. The volume to be injected contains 50 ng of base/neutral and 50 ng of acid surrogates (assuming 100% recovery).
3	The recommended sequence for a 20-sample batch is as follows:

1	DFTPP Tuning Criteria
2	DDT Breakdown Percentage
3	Benzidine and Pentachlorophenol Tailing Factors
4	CCV
5	Method Blank
6	LCS
7	Matrix Spike
8	Matrix Spike
9	Samples 1-20

4	If the response for any quantitation ion exceeds the initial calibration range of the GC/MS system, the sample extract must be diluted and reanalyzed in the upper half of the calibration range. Additional internal standard must be added to the diluted extract to maintain the same concentration as in the calibration standards (40 µg/mL, unless a more sensitive GC/MS system is being used, e. g., 4 µg/mL for SIM).
5	Evaluate the specific internal standard response. Dilutions may be required to meet this criterion. Notes: Specific analytes associated with an internal standard within -50 to +100% from the last calibration verification (CCV) may be reported with approval from the supervisor or manager even if other internal standards in that analysis are outside limits. Only analytes associated with the internal standard(s) within limits may be reported from that analysis.
6	The use of selected ion monitoring (SIM) is acceptable for applications requiring detection

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	limits below the normal range of electron impact mass spectrometry. Multiple ions are used for compound identification; see Attachment 2. Secondary ions may drop below 30% relative intensity at concentrations less than 1 µg/mL.

10.5 Qualitative analysis

- The qualitative identification of compounds determined by this method is based on retention time and on comparison of the sample mass spectrum, after background correction, with characteristic ions in a reference mass spectrum. The reference mass spectrum must be kept up to date and obtained through analysis of known standards on the instrument using the conditions of this method. The characteristic ions from the reference mass spectrum are defined as the three ions of greatest relative intensity, or any ions over 30% relative intensity, if less than three such ions occur in the reference spectrum. Attachments 1 and 2 list the primary and secondary ions for each analyte. Compounds are identified when the following criteria are met.
- The intensities of the characteristic ions of a compound must maximize in the same scan or within one scan of each other. Selection of a peak by a data system target compound search routine where the search is based on the presence of a target chromatographic peak containing ions specific for the target compound at a compound-specific retention time is accepted as meeting this criterion.
- The RRT of the sample component is within ± 0.06 RRT units of the RRT of the standard component.
- The relative intensities of the characteristic ions agree within 30% of the relative intensities of these ions in the reference spectrum. **Example: For an ion with an abundance of 50% in the reference spectrum, the corresponding abundance in a sample spectrum can range between 20% and 80%.** When two or more analytes that co-elute share secondary ions, and all the characteristic secondary ions for the target analyte are present but outside the ±30% relative intensity, the compound is reported as positive if there is no interference with the primary quantitation ion. If co-eluting peaks share the primary ion, the analyte may only be reported as a co-eluting pair. (See Attachment 1.)
- Identification is hampered when sample components are not resolved chromatographically and produce mass spectra containing ions contributed by more than one analyte. When gas chromatographic peaks obviously represent more than one sample component (i. e., a broadened peak with shoulder(s) or a valley between two or more maxima), appropriate selection of analyte spectra and background spectra is important.
- Examination of extracted ion current profiles of appropriate ions can aid in the selection of spectra and in qualitative identification of compounds. When analyses co-elute (i. e., only one chromatographic peak is apparent), the identification criteria may be met, but each analyte spectrum contains extraneous ions contributed by the co-eluting compound. The analyst must carefully weigh the background spectrum and the spectrum of any co-eluting analytes whenever assessing a potential hit. Analyst experience in interpreting mass spectral data and the above specified guidelines are used together to interpret difficult matrices. If all of the ions associate with the reference spectrum for the target analyte are present and within the ±30% criteria, a positive result is assumed even in the presence of extraneous ion fragments without presumptive evidence (all ions associated with the target analyte are also present in the interfering peak) for a negative identification.
- Structural isomers that produce very similar mass spectra are identified as individual isomers if they have sufficiently different GC retention times. Sufficient GC resolution is achieved if the height of the valley between two isomer peaks is less than 25% of the sum of the two peak heights. Otherwise, structural isomers are identified as isomeric pairs. (See Attachment 1.)
- For samples containing components not associated with the calibration standards or the requested target list, a library search may be made for the purpose of tentative identification.

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The necessity to perform this type of identification will be determined by the purpose of the analyses being conducted. Data system library search routines do not use normalization routines that would misrepresent the library or unknown spectra when compared to each other.

- For example, the RCRA permit or waste delisting requirements may require the reporting of non-target analytes. Only after visual comparison of sample spectra with the nearest library searches may the analyst assign a tentative identification. Guidelines for tentative identification are:
  1) Relative intensities of major ions in the reference spectrum (ions >10% of the most abundant ion) are present in the sample spectrum.
  2) The relative intensities of the major ions agree within ±20%. **Example: For an ion with an abundance of 50% in the standard spectrum, the corresponding sample ion abundance must be between 30 and 70%.**
  3) Molecular ions present in the reference spectrum are present in the sample spectrum.
  4) Ions present in the sample spectrum but not in the reference spectrum are reviewed for possible background contamination or presence of co-eluting compounds.
  5) Ions present in the reference spectrum but not in the sample spectrum are reviewed for possible subtraction from the sample spectrum because of background contamination or co-eluting peaks. Data system library reduction programs can sometimes create these discrepancies.

10.6 Quantitative analysis

- Once a compound has been identified, the quantitation of that compound is based on the integrated abundance of the primary characteristic ion from the EICP.
- If the RSD of a compound's response factor is 15% or less, then the concentration in the extract is determined using the average response factor (RF) from initial calibration data. If greater than 15%, use linear regression.
- Where applicable, the concentration of any non-target compounds identified in the sample is estimated. The same formulae are used with the following modifications: The areas A_x and A_t are from the total ion chromatograms, and the RF for the compound is assumed to be 1.
- The resulting concentration is reported indicating: (1) that the value is an estimate, and (2) which internal standard was used to determine concentration. Use the nearest internal standard free of interferences.
- Structural isomers that produce very similar mass spectra are quantitated as individual isomers if they have sufficient GC retention times. Sufficient GC resolution is achieved if the height of the valley between two isomer peaks is less than 25% of the sum of the two peak heights. Otherwise, structural isomers are quantitated as isomeric pairs.

10.7 Instrument Maintenance

Careful examination of the standard chromatogram indicates whether the column is still performing acceptably, the injector is leaking, the injector septum needs replacing, etc. Re-calibration of the instrument must take place when the performance changes to the point that the calibration verification acceptance criteria cannot be achieved. In addition, significant maintenance activities or hardware changes may also require re-calibration. These significant maintenance activities include, changing, replacing, or reversing the column; cleaning the MS source; changing the electron multiplier; or injector port.

11.0 Calculations / Data Reduction

11.1 Accuracy

$$\text{\% Recovery} = \frac{\text{Measured concentration}}{} \times 100$$

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Known concentration

11.2 Precision (RPD)

RPD = $\frac{\text{Absolute value (orig. sample value - dup. sample value)}}{\text{(Orig. sample value + dup. sample value)/2}} \times 100$

11.3 Breakdown Calculation:

% Breakdown of DDT = $\frac{\text{Sum of degradation peak areas (DDD + DDE)}}{\text{Sum of all peak areas (DDT + DDE + DDD)}} \times 100$

1.4 Response Factor

$$RF = \frac{A_s x C_{is}}{A_{is} x C_s}$$

A_s = Peak area of the analyte or surrogate.
A_{is} = Peak area of the internal standard.
C_s = Concentration of the analyte or surrogate, in µg/L.
C_{is} = Concentration of the internal standard, in µg/L.

11.5 Mean Response Factor, Standard Deviation, Relative Standard Deviation

$$\mathbf{RF_{mean}} = \frac{\sum_{i=1}^{n} RF_i}{n}$$

$$\mathbf{SD} = \frac{\sum_{i=1}^{n} \left(RF_i - RF_{mean}\right)^2}{n-1}$$

RSD = $\frac{\text{SD x 100}}{\text{RF}_{mean}}$

11.6 % Difference, % Drift

% Difference = $\frac{(RF_v) - (\text{Avg. RF})}{\text{(Avg. RF)}} \times 100$

RF_v = RF from verification standard
Avg. RF = Average RF from Initial Calibration.

% Drift = $\frac{\text{Result - True Value}}{\text{True Value}} \times 100$

11.7 Linear Calibration Using a Least Squares Regression

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This approach is not used for analytes that meet the RSD limits. For calibration, x is the mass of the analyte in the sample aliquot introduced into the instrument and y is the area (or height) or the response, as in:

$x = C_s$ and $y = A_s$

A linear least squares regression attempts to construct a linear equation of the form:

$y = ax + b$

by minimizing the differences between the observed results (y_i, the instrument response) and the predicted results (y_i', the response calculated from the constructed equation). The regression equation is:

$y_i' = ax_i + b$

a = regression coefficient or the slope of the line.
b = the y-intercept.
y_i' = predicted (or calculated) response for the i^{th} calibration standard.
x_i = mass of analyte in the i^{th} calibration standard aliquot introduced into the instrument.

The sum of the squares of the differences is minimized to obtain a and b:

$$\sum_{i=1}^{n}(y_i - y_i')^2$$

n = total number of calibration points. The regression calculations attempt to minimize this sum of the squares, hence the name "least squares regression."

Weighting the sum of the square of the differences may significantly improve the ability of the least squares regression to fit the linear model to the data. The general form of the sum of the squares of the differences containing the weighting factor is:

$$\sum_{i=1}^{n} w_i(y_i - y_i')^2$$

w_i = weighting factor for the i^{th} calibration standard (w=1 for unweighted least squares regression).
y_i – observed instrument response (area) for the i^{th} calibration standard.
y_i' = predicted (or calculated) response for the i^{th} calibration standard.
n = total number of calibration standards.

The mathematics used in least squares regression has a tendency to favor numbers of larger value over numbers of smaller value. Thus the regression curves that are generated tend to fit points that are at the upper calibration levels better than those points at the lower calibration levels. To compensate for this, a weighting factor which reduces this tendency can be used. Examples of allowed weighting factors which can place more emphasis on numbers of smaller value are:

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$w_i - 1/y_i$ or $w_i = 1/y_i^2$

Do not include the origin (0, 0) as an extra calibration point.

11.8 Coefficient of Determination

$$r^2 = \frac{\left(\sum xy\right)^2}{\sum x^2 \sum y^2}$$

Correlation Coefficient

$$r = \frac{\left(\sum xy\right)}{\sqrt{\sum x^2 \sum y^2}}$$

y = Response or Response ratio
x = Concentration

11.9 Calculation

- For **aqueous** samples:

$$\textbf{Concentration (µg/L)} = \frac{A_x V_t D}{RF_{mean} V_s} \quad \text{or} \quad \frac{(\mu g/mL \text{ from instrument})(D)(1000)}{mL \text{ extracted}}$$

A_x = Area of the peak for the analyte in the sample.
V_t = Total volume of the concentrated extract (mL).
D = Dilution factor, if the sample or extract was diluted prior to analysis. If no dilution was made, D = 1. The dilution factor is always dimensionless.
RF_{mean} = Mean response factor from the initial calibration (area/concentration).
V_s = Volume of the aqueous sample extracted in mL.

- For **non-aqueous** samples:

$$\textbf{Concentration (µg/kg)} = \frac{A_x V_t D}{RF_{mean} W_s} \quad \text{or} \quad \frac{(\mu g/mL \text{ from instrument})(D)(1000)}{g \text{ extracted}}$$

A_x, V_t, D, RF_{mean} are the same as for aqueous samples, and
W_s = Weight of sample extracted (g). The wet weight or dry weight may be used, depending upon the specific application of the data.

12.0 Method Performance

12.1 Method Detection Limit Study (MDL): The method detection limit (MDL) is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. The MDL is determined according to the laboratory's MDL procedure in SOP Determination of Method Detection Limits / NV08-202. MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix, and may not be achievable in all environmental matrices. The laboratory maintains MDL studies for analyses performed; these are verified at least annually unless method requirements require a greater frequency.

12.2 Demonstration of Capability: The laboratory demonstrates initial proficiency by generating data of acceptable accuracy and precision for target analyses in a clean matrix. The laboratory also repeats the operation whenever new staff is trained or significant changes in instrumentation are made and on an annual basis thereafter. See the training section of TestAmerica-Nashville's QA Manual and SOP Training / NV08-199 for information on how to accomplish this demonstration.

12.3 Training Requirements: Demonstration of Capability is performed initially when learning the method and annually thereafter. Four Laboratory Control Samples resulting in an average %

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recovery within the control limits and a precision less than the quality control maximum are required.

12.4 Proficiency Testing Studies: The laboratory participates in formal proficiency testing (PT) studies, where solutions of unknown concentrations are analyzed and the performance of all participants is compared. See the QA department for the results of recent PT studies.

13.0 Pollution Control

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i. e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention."

14.0 Waste Management

Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes must be stored, managed, and disposed of in accordance with all federal and state laws and regulations. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to the QA Manual and SOP Waste Disposal / NV10-83.

15.0 References / Cross References

15.1 Method 8270C, SW-846 Update III Revision 3, December 1996 and Method 8270D, Update IV, Revision 4, February 2007.
15.2 Method 8000B, SW-846, Revision 2, December 1996, Method 8000C, Revision 3, March 2003.
15.3 TestAmerica Nashville's Quality Assurance Manual.
15.4 TestAmerica Nashville's Control Limits Manual.
15.5 Corporate Environmental Health and Safety Manual (CW-E-M-001).
15.6 SOPs: Manual Integration / CA-Q-S-002, Selection of Calibration Points / CA-P-T-002, Waste Disposal / NV10-83, Training Procedures for Environmental Technical Staff / NV08-199, Determination of Method Detection Limits / NV08-202, Reagent and Standard Purchase / NV08-214, 3550 / NV03-23, and 3510 / NV03-24, 3541 / NV03-231, 3580 / NV03-106.

16.0 Method Modifications

Item	Modification
1	See Attachment 5 for the State of Ohio specific criteria.
2	See Attachment 6 for the State of Missouri DRO, CA LUFT DRO by GC/MS.
3	Verify with state certifications the correct version of this method to report.
4	SIM is not allowed for South Carolina samples unless pre-approved by the state on a project-specific basis.

17.0 Attachments

Attachment 1, Characteristic Ions for Semivolatile Compounds[a]

Compound	Retention Time (minutes)	Primary Ion	Secondary Ion(s)
1,4-Dioxane	2.568	88	58
n-Nitrosodimethylamine	2.700	74	42, 44
Pyridine	2.714	79	52
2-Picoline	3.464	93	66, 92
n-Nitrosomethylethylamine	3.558	88	42, 43, 56

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Compound	Retention Time (minutes)	Primary Ion	Secondary Ion(s)
2-Fluorophenol (surr)	3.687	112	64
Methyl methanesulfonate	3.764	80	79, 65, 95
n-Nitrosodiethylamine	4.009	102	42, 57, 44, 56
Ethyl methanesulfonate	4.197	79	109, 97, 45, 65
Hexachloropropene	4.261	213	211,215,117,106,141
Phenol-d_5 (surr)	4.266	99	42, 71
Aniline	4.270	93	66, 65
Bis(2-chloroethyl) ether	4.294	93	63, 95
Phenol	4.275	94	65, 66
2-Chlorophenol	4.345	128	64, 130
1,3-Dichlorobenzene	4.425	146	148, 113
1,4-Dichlorobenzene-d_4 (IS)	4.444	152	150, 115
1,4-Dichlorobenzene	4.454	146	148, 113
Pentachloroethane	4.474	117	165, 167, 119
Benzyl alcohol	4.543	79	108, 77
n-Decane	4.550	57	
1,2-Dichlorobenzene	4.571	146	148, 113
2-Methylphenol	4.628	108	107, 77, 79, 90
Bis(2-chloroisopropyl) ether	4.632	45	77, 79
N-Nitrosodi-n-propylamine	4.717	130	42, 101, 70
3, 4-Methylphenol	4.717	107	108, 77, 79, 90
Hexachloroethane	4.764	117	201, 199
Nitrobenzene-d_5 (surr)	4.806	82	128, 54
Nitrobenzene	4.816	77	123, 65
n-Nitrosopyrrolidine	4.907	102	41, 42, 68, 69
Acetophenone	4.912	105	71, 51, 120
n-Nitrosomorpholine	4.916	108	116, 86
o-Toluidine	4.940	106	107, 77, 51, 79
Isophorone	4.957	82	95, 138
2-Nitrophenol	5.018	139	109, 65
2,4-Dimethylphenol	5.037	122	107, 121
Bis(2-chloroethoxy)methane	5.088	93	95, 123
n-Nitrosopiperidine	5.114	114	42, 55, 56, 41
Benzoic acid	5.116	105	122, 77
2,4-Dichlorophenol	5.168	162	164, 98
1,2,4-Trichlorobenzene	5.215	180	182, 145
Naphthalene-d_8 (IS)	5.248	136	68
Naphthalene	5.257	128	129, 127
o,o,o-Triethylphosphorthioate	5.302	198	80, 53, 54164, 63
4-Chloroaniline	5.304	127	129, 65, 92
Hexachlorobutadiene	5.370	225	223, 227
a,a-Dimethylphenethylamine	5.372	58	91, 65, 134, 42
2,6-Dichlorophenol	5.523	162	
Hexachloropropene	5.556	213	211, 215, 117, 106, 141
4-Chloro-3-methylphenol	5.615	142	107, 144
2-Methylnaphthalene	5.704	142	141
n-Nitrosodi-n-butylamine	5.729	84	57, 41, 116, 158
1,4-Phenylenediamine	5.734	198	80, 53, 54, 52
1-Methylnaphthalene	5.779	142	141, 115
Hexachlorocyclopentadiene	5.854	237	235, 272
Isosafrole (trans)	5.861	162	131, 104, 77

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Compound	Retention Time (minutes)	Primary Ion	Secondary Ion(s)
2,4,6-Trichlorophenol	5.911	196	198, 200
2,4,5-Trichlorophenol	5.944	196	198, 97, 132, 99
2-Fluorobiphenyl (surr)	5.953	172	171
2-Chloronaphthalene	6.033	162	127, 164
Isosafrole (cis)	6.054	162	131, 104, 77
1,2,4,5-Tetrachlorobenzene	6.063	216	214,179,108,143,218
2-Nitroaniline	6.118	138	92, 65
2,3-Dichloroaniline	6.134	161	90, 63
Safrole	6.204	162	104, 77, 103, 135
Dimethyl phthalate	6.245	163	194, 164
1-Chloronaphthalene	6.284	162	127, 164
2,6-Dinitrotoluene	6.296	165	63,89, 121
Acenaphthylene	6.320	152	151, 153
1,4-Naphthoquinone	6.374	158	104, 102, 76, 50, 130
3-Nitroaniline	6.404	138	108, 92
Acenaphthene	6.447	154	153, 152
2,4-Dinitrophenol	6.470	184	63, 154
1,3-Dinitrobenzene	6.486	168	76, 50, 75, 92, 122
4-Nitrophenol	6.527	65	109, 139
Dibenzofuran	6.560	168	139
2,4-Dinitrotoluene	6.574	165	63, 89, 182
Acenaphthene-d_{10} (IS)	6.656	164	162, 160
Diethyl phthalate	6.738	149	177, 150
4-Chlorophenyl phenyl ether	6.790	204	206, 141
Fluorene	6.799	166	165,167
Pentachlorobenzene	6.806	250	252,108,248,215,254
4-Nitroaniline	6.837	138	65, 108, 92, 80, 39
1-Naphthylamine	6.844	143	115, 89, 63
4,6-Dinitro-2-methylphenol	6.865	198	51, 105, 182, 77
n-Nitrosodiphenylamine	6.879	169	168, 167
2-Naphthylamine	6.895	143	115, 116
2,3,4,6-Tetrachlorophenol	6.900	232	131,230,166,234,168
1,2-Diphenylhydrazine	6.903	77	105, 182
2,4,6-Tribromophenol (surr)	6.987	330	332, 141
Thionazine	7.027	107	96, 97,143, 79, 68
5-Nitro-o-toluidine	7.051	152	77, 79, 106, 94
Diphenylamine	7.107	168	169, 167
4-Bromophenyl phenyl ether	7.138	248	250, 141
Hexachlorobenzene	7.255	284	142, 249
Sulfotepp	7.276	322	97, 202
1,3,5-Trinitrobenzene	7.314	213	74, 120, 91, 63
Diallate (trans)	7.337	86	234, 43, 70
Phenacetin	7.337	108	180,179,109,137,80
Phorate	7.347	75	121, 97, 93, 260
Pentachlorophenol	7.387	266	264, 268
Diallate (cis)	7.403	86	234, 43, 70
Dimethoate	7.474	87	93, 125, 143, 229
Phenanthrene-d_{10} (IS)	7.476	188	94, 80
Phenanthrene	7.495	178	179, 176
Anthracene	7.528	178	176, 179
4-Aminobiphenyl	7.568	169	168, 170, 115
n-Octadecane	7.586	58	71, 85

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Compound	Retention Time (minutes)	Primary Ion	Secondary Ion(s)
Pronamide	7.619	173	175, 145, 109, 147
Carbazole	7.641	167	139, 84
Pentachloronitrobenzene	7.676	237	142,214,249,295,265
Disulfoton	7.723	88	97, 89, 142, 186
Dinoseb	7.737	211	163, 147, 117, 240
Di-n-butyl phthalate	7.914	149	150, 104
Methyl parathion	8.000	109	125, 263, 79, 93
Parathion	8.292	109	97, 291, 139, 155
4-Nitroquinoline-1-oxide	8.310	190	101, 128, 75, 116
Methapyrilene	8.371	58	50, 191, 71
Fluoranthene	8.374	202	100, 101, 203
Benzidine	8.464	184	92, 185
Isodrin	8.522	193	66, 195, 263,265,147
Pyrene	8.543	202	100, 101, 200, 203
Terphenyl-d_4 (surr)	8.652	244	122, 212
Aramite	8.870	191	319, 334, 197, 321
Dimethylaminoazobenzene	9.001	120	77, 105, 148, 42
Butyl benzyl phthalate	9.028	149	91, 206
Chlorobenzilate	9.034	139	253, 111, 141
Hexachlorophene	9.070	185	209,406
3,3'-Dimethylbenzidine	9.251	212	106, 196, 180
Bis (2-ethylhexyl) adipate	9.298	129	57, 112, 147
4,4'-Methylenebis(2-chloroaniline)	9.301	231	266, 140, 77
Kepone	9.316	272	274,237,178,143,270
3,3'-Dichlorobenzidine	9.423	252	254, 126
Benz(a)anthracene	9.446	228	229, 226
2-Acetylaminofluorene	9.453	181	180, 223, 152
Chrysene-d_{12} (IS)	9.456	240	120, 36
Chrysene	9.474	228	226, 229
Bis(2-ethylhexyl) phthalate	9.474	149	167, 279
Di-n-octyl phthalate	9.926	149	167, 43
Benzo(b)fluoranthene	10.292	252	253, 125
3-Methylcholanthrene	11.305	268	252,253,126,134,113
Benzo(k)fluoranthene	10.311	252	253, 125
Benzo(a)pyrene	10.579	252	253, 125
7,12-Dimethylbenz(a)anthracene	10.600	256	241, 239, 120
Perylene-d_{12} (IS)	10.631	264	260, 265
Indeno)1,2,3-c,d)pyrene	11.778	276	138, 277
Dibenz(a,h)anthracene	11.783	278	139, 279
Dibenz(a,j)acridine	11.987	279	280, 277, 250
Dibenz(a,j)acridine	11.987	279	280, 277, 250
Benzo(g,h,i)perylene	12.107	276	138, 277
IS = internal standard			
surr = surrogate			
[a]See Attachment 2 for Retention Times and Ions used with SIM.			

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Attachment 2, Characteristic Ions for PAH Compounds Using SIM

Compounds	RT	Primary	Secondary*
1,4-Dichlorobenzene-d_4	6.66	152	
2-Fluorophenol	5.471	112	64
Phenol-d_5	6.251	99	71.1
Naphthalene-d_8	8.24	136	
Nitrobenzene-d_5	7.32	82.1	128.1
Naphthalene	8.27	128.1	129.1
2-Methylnaphthalene	9.13	142.1	141.1
1-Methylnaphthalene	9.42	142.1	141.1
Acenaphthene-d_{10}	10.91	164.1	
2-Fluorobiphenyl	9.82	172.1	
Acenaphthylene	10.67	152.1	151.1
Acenaphthene	10.958	15.1	154.1
Fluorene	11.7	166.1	167.1
Phenanthrene-d_{10}	13.3	188	
2,4,6-Tribromophenol	12.167	329.8	331.8
Phenanthrene	13.33	178.2	176.2
Anthracene	13.4	178.2	176.2
Fluoranthene	15.28	202.2	101.1
Chrysene-d_{12}	17.64	240.1	
Pyrene	15.66	202.2	101.1
Terphenyl-d_{14}	15.89	244.2	
Benzo(a)anthracene	17.61	228.2	229.2
Chrysene	17.68	228.2	229.2
Perylene-d_{12}	20.2	264.2	
Benzo(b)fluoranthene	19.45	252.2	126.1
Benzo(k)fluoranthene	19.49	252.2	126.1
Benzo(a)pyrene	20.08	252.2	126.1
Indeno(1,2,3-cd)pyrene	22.69	276.2	277.2
Dibenzo(ah)anthracene	22.7	278.2	279.2
Benzo(ghi)perylene	23.43	276.2	277.2
Internal standards are in **bold.**			

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Attachment 3, SIM Mass Groups

Mass Group	Compound	RT	Primary	Secondary	Dwell Time
1	2-Fluorophenol	5.42	112	64	25 ms
	Phenol-d_5	6.2	99	71.1	
	1,4-Dichlorobenzene-d_4	6.6	152		
	Nitrobenzene-d_5	7.26	82.1	128.1	
4.20 min.					
2	Naphthalene-d_8	8.17	136		50 ms
	Naphthalene	8.2	128.1	129.1	
	2-Methylnaphthalene	9.19	142.1	141.1	
	1-Methylnaphthalene	9.35	142.1	141.1	
	2-Fluorobyphenyl	9.75	172.1		
5.35 min.					
3	Acenaphthylene	10.6	152.1	151.1	25 ms
	Acenaphthalene-d_{10}	10.83	164.1		
	Acenaphthene	10.88	153.1	154.1	

Mass Group	Compound	RT	Primary	Secondary	Dwell Time
	Fluorene	11.69	166.1	167.1	
	2,4,6-Tribromophenol	12.11	329.8	331.8	
6.55 min.					
4	Phenanthrene-d_{10}	13.21	188		50 ms
	Phenanthrene	13.25	178.2	176.2	
	Anthracene	13.32	178.2	176.2	
	Fluoranthene	15.21	202.2	101.1	
	Pyrene	15.58	202.2	101.1	
	Terphenyl-d_{14}	15.81	244.2		
7.75 min.					
5	Benzo(a)anthracene	17.52	228.2	229.2	100 ms
	Chrysene-d_{12}	17.55	240.1		
	Chrysene	17.59	228.2	229.2	
9.85 min.					
6	Benzo(b)fluoranthene	19.34	252.2	126.1	100 ms
	Benzo(k)fluoranthene	19.38	252.2	126.1	
	Benzo(a)pyrene	19.96	252.2	126.1	
	Perylene-d_{12}	20.07	264.2		
10.65 min.					
7	Indeno(1,2,3-cd)pyrene	22.5	276.2	277.2	100 ms
	Dibenzo(a,h)anthracene	22.52	278.2	279.2	
	Benzo(g,h,i)perylene	23.21	276.2	277.2	
12.20 min.					

Attachment 4, 8270D Minimum Response Factor Criteria for Initial and Continuing Calibration Verification Using the Suggested Ions from Attachments 1 and 2.

Compound	Minimum RF	Compound	Minimum RF
Benzaldehyde	0.010	4-Nitrophenol	0.010
Phenol	0.800	Dibenzofuran	0.800
Bis(2-chloroethyl)ether	0.700	2,4-Dinitrotoluene	0.200
2-Chlorophenol	0.800	Diethyl phthalate	0.010
2-Methylphenol	0.700	1,2,4,5-tetrachlorobenzene	0.010
2,2'-Oxybis-(1-chloropropane)	0.010	4-Chlorophenyl-phenyl ether	0.400
Acetophenone	0.010	Fluorene	0.900
4-Methylphenol	0.600	4-Nitroaniline	0.010
n-Nitroso-di-n-propylamine	0.500	4,6-Dinitro-2-methylphenol	0.010
Hexachloroethane	0.300	4-Bromophenyl-phenyl ether	0.100
Nitrobenzene	0.200	n-Nitrosodiphenylamine	0.010
Isophorone	0.400	Hexachlorobenzene	0.100
2-Nitrophenol	0.100	Atrazine	0.010
2, 4-Dimethylphenol	0.200	Pentachlorophenol	0.050
Bis(2-chloroethoxy)methane	0.300	Phenanthrene	0.700
2,4-Dichlorophenol	0.200	Anthracene	0.700
Naphthalene	0.700	Carbazole	0.010
4-Chloroaniline	0.010	Di-n-butyl phthalate	0.010
Hexachlorobutadiene	0.010	Fluoranthene	0.600
Caprolactam	0.010	Pyrene	0.600
4-Chloro-3-methylphenol	0.200	Butyl benzyl phthalate	0.010
2-Methylnaphthalene	0.400	3,3'-Dichlorobenzidine	0.010

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Compound	Minimum RF	Compound	Minimum RF
Hexachlorocyclopentadiene	0.050	Benzo(a)anthracene	0.800
2,4,6-Trichlorophenol	0.200	Chrysene	0.700
2,4,5-Trichlorophenol	0.200	Bis-(2-ethylhexyl)phthalate	0.010
1,1'-Biphenyl	0.010	Di-n-octyl phthalate	0.010
2-Chloronaphthalene	0.800	Benzo(b)fluoranthene	0.700
2-Nitroaniline	0.010	Benzo(k)fluoranthene	0.700
Dimethyl phthalate	0.010	Benzo(a)pyrene	0.700
2,6-Dinitrotoluene	0.200	Indeno(1,2,3-cd)pyrene	0.500
Acenaphthylene	0.900	Dibenz(a,h)anthracene	0.400
3-Nitroaniline	0.010	Benzo(g,h,i)perylene	0.500
Acenaphthene	0.900	2,3,4,6-Tetrachlorophenol	0.010
2,4-Dinitrophenol	0.010		

Attachment 5, State of Ohio Specific Criteria.
Only those compounds in the original EPA Method 8270C may be reported. Any compounds in this SOP in italics in Section 1 are not part of the original 8270C method.

Attachment 6, Missouri Department of Natural Resources (and CA LUFT) require(s) that **DRO** be analyzed by GC/MS.

- Tuning and frequency requirements are the same as in 8270, omitting DDT, Pentachlorophenol, and Benzidine.
- Extract water samples per SOP 3510 / SA03-24 and solid samples per SOP 3550 / SA03-23.
- Only base/neutral surrogates are needed.
- GC/MS mass range is 35-550 ηmu.
- Use a five-point calibration curve with 1:1 unleaded gasoline and #2 diesel fuel at 1,000 µg/mL each in Methylene chloride.
- Retention time windows set using C_{10}, C_{21}, and C_{35}. For DRO, set RT 0.1 minutes <u>after</u> C10 to 0.1 minutes <u>after</u> C21. For ORO, set RT 0.1 minutes <u>after</u> C_{21} to 0.1 minutes <u>after</u> C_{35}. Verify RT daily (24 hours) by running component standard.
- Quantitative using baseline-to-baseline, not valley-to-valley. The Total Ion Chromatogram must be used to quantitate.
- The Response Factor determined for DRO (C_{10}-C_{21}) <u>must</u> be used for C_{21}-C_{35}.
- Subtract area from any Internal Standard and surrogates.
- % RSD ≤ 20.
- Run a CCV every 12 hours, must contain all targets reported, at mid-point of calibration, % D ≤ 20.
- Run a Method Blank every extraction batch, and LCS and MS/MSD per batch.
- May reprocess file to quantitate PAH if needed. For individual targets, % RSD ≤ 15.
- Quantitation of DRO must be by external standard.

18.0 <u>Revision History</u>

- Revision 12, 22 October 2008
  - Integration for TestAmerica and STL operations.
  - Insert corrective action procedures
  - To incorporate Update IV criteria.
- Revision 13, 9 October 2009
  - Consolidation of text, general editing.
  - Add Appendix IX and miscellaneous compound details.
  - Distinguish 8270C versus 8270D.

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Title: VOLATILE ORGANIC COMPOUNDS BY GAS CHROMATOGRAPHY/MASS SPECTROMETRY (GC/MS) SW-846 METHOD 8260B

Approvals (Signature/Date):

Glenn L. Norton 9-13-10
Glenn Norton Date
Department Operations Manager

John Haley 9-13-10
John Haley Date
Department Supervisor

Michael H. Dunn 9-17-10
Michael H. Dunn Date
Technical Director

9-22-10
Jared Knierim Date
Health & Safety Manager / Coordinator

E S Smith 9/27/10
Eric S. Smith Date
Quality Assurance Director

J A Mitchell 9.29.10
Johnny A. Mitchell Date
Laboratory Director

For NELAP purposes, this SOP is known as 8260 / **SA**05-77. The new NV designation derives from the integration of TestAmerica and STL.

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1.0 Scope and Application

1.1 Analyte, Matrices: This method is used to determine volatile organic compounds in a variety of matrices; it is applicable to nearly all types of samples, regardless of water content, ground and surface water, aqueous sludges, caustic liquors, acid liquors, waste solvents, oily wastes, mousses, tars, fibrous wastes, polymeric emulsions, filter cakes, spent carbons, spent catalysts, soils, and sediments. The following compounds can be determined by this method:

Compound	CAS No. (a)	Compound	CAS No. (a)
Acetone[1, 2, 5, 6, 7]	67-64-1	1,4-Dioxane[2, 8]	123-91-1
Acetonitrile[2, 7]	75-05-8	Ethanol[3]	64-17-5
Acrolein (Propenal)[2, 7]	107-02-8	Ethyl acetate[4]	141-78-6
Acrylonitrile[2, 6, 7]	107-13-1	Ethylbenzene[1, 2, 5, 6, 7, 8, 9]	100-41-4
Allyl chloride[2, 7]	107-05-1	Ethyl methacrylate[2, 7]	97-63-2
Benzene[1, 2, 5, 6, 7, 8, 9]	71-43-2	Ethyl-tert-butyl-ether (ETBE)[3]	637-92-3
Benzyl chloride[4]	100-44-7	Heptane[4]	142-82-5
Bromobenzene[1]	108-86-1	Hexachlorobutadiene[1, 2]	87-68-3
Bromochloromethane[1, 6, 7]	74-97-5	Hexane[4]	110-54-3
Bromodichloromethane[1,2, 6, 7]	75-27-4	2-Hexanone[1, 2, 5, 6, 7]	591-78-6
Bromoethane[4]	74-96-4	Iodomethane[2, 6, 7]	74-88-4
Bromoform[1, 2, 5, 6, 7]	75-25-2	Isobutyl alcohol[2, 7]	78-83-1
Bromomethane[1, 2, 5, 6, 7]	74-83-9	Isobutylisobutyrate[4]	97-85-8
2-Butanone (MEK)[1, 2, 5, 6, 7, 8]	78-93-3	Isopropy alcohol[4]	67-63-0
Butyl acetate	123-86-4	Isopropylbenzene[1, 5, 9]	98-82-8
n-Butyl Alcohol[4]	71-36-3	Isopropylether (IPE)[3]	180-20-3
t-Butyl Alcohol[3]	75-65-0	p-Isopropyltoluene[1, 9]	99-87-6
n-Butylbenzene[1, 9]	104-51-8	Methacrylonitrile[2, 7]	126-98-7
sec-Butylbenzene[1, 9]	135-98-8	Methyl acetate[5]	79-20-9
tert-Butylbenzene[1, 9]	98-06-6	Methylcyclohexane[5]	108-87-2
Carbon disulfide[1, 2, 5, 6, 7, 8]	75-15-0	Methylene chloride[1, 2, 5, 6, 7]	75-09-2
Carbon tetrachloride[1, 2, 5, 6, 7]	56-23-5	Methyl methacrylate[2, 7]	80-62-6
Chlorobenzene[1, 2, 5, 6, 7, 8]	108-90-7	1-Methylnaphthalene[4]	90-12-0
Chloroethane[1, 2, 5, 6, 7]	75-00-3	2-Methylnapthalene[4]	91-57-6
2-Chloroethyl vinylether[4]	110-75-8	4-Methyl-2-pentanone (MIBK)[1, 2, 5, 6, 7]	108-10-1
Chloroform[1, 2, 5, 6, 7, 8]	67-66-3	Methyl-t-butyl ether[1, 3, 4, 5, 9]	1634-04-4
1-Chlorohexane[4]	544-10-5	Naphthalene[1, 2, 9]	91-20-3
Chloromethane[1, 2, 5, 6, 7]	74-87-3	2-Nitropropane[4]	79-46-9
Chloroprene[2, 7]	126-99-8	Nonane[4]	111-84-2
2-Chlorotoluene[1]	95-49-8	Octane[4]	111-65-9
4-Chlorotoluene[1]	106-43-4	Pentane[4]	109-66-0
Cyclohexanone[4]	108-94-1	Propionitrile[2, 7]	107-12-0
Cyclohexane[4, 5]	110-82-7	n-Propylbenzene[1, 9]	103-65-1
Dibromochloromethane[1, 2, 5, 6, 7]	124-48-1	Styrene[1, 2, 5, 6, 7]	100-42-5
1,2-Dibromo-3-chloropropane[1, 2, 5, 7]	96-12-8	tert-butyl-formate (TBF)[3]	762-75-4
1,2-Dibromoethane[1, 2, 5, 8]	106-93-4	tert-Amyl-alcohol (TAA)[3]	75-85-4
Dibromomethane[1, 2, 6, 7]	74-95-3	tert-Amyl-methyl-ether (TAME)[3]	994-05-8
2,6-Dimethyl-4-heptanone(DIBK)[4]	108-83-8	1,1,1,2-Tetrachloroethane[1, 2, 6, 7]	630-20-6
1,2-Dichlorobenzene[1, 2, 5, 6, 7]	95-50-1	1,1,2,2-Tetrachloroethane[1, 2, 5, 6, 7]	79-34-5
1,3-Dichlorobenzene[1, 2, 5, 7]	541-73-1	Tetrachloroethene[1, 2, 5, 6, 7]	127-18-4
1,4-Dichlorobenzene[1, 2, 5, 6, 7]	106-46-7	Tetrahydrofuran[4]	109-99-9
cis-1,4-Dichloro-2-butene[4]	1476-11-5	Toluene[1, 2, 5, 6, 7, 8, 9]	108-88-3
trans-1,4-Dichloro-2-butene[2, 6, 7]	110-57-6	1,2,3-Trichlorobenzene[1]	87-61-6

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Compound	CAS No. (a)	Compound	CAS No. (a)
Dichlorodifluoromethane[1, 2, 5, 7]	75-71-8	1,2,4-Trichlorobenzene[1, 2, 5]	120-82-1
1,1-Dichloroethane[1, 2, 5, 6, 7]	75-34-3	1,3,5-Trichlobenzene[4]	176-02-8
1,2-Dichloroethane[1, 2, 5, 6, 7, 8]	107-06-2	1,1,1-Trichloroethane[1, 2, 5, 6]	71-55-6
1,1-Dichloroethene[1, 2, 5, 6, 7]	75-35-4	1,1,2-Trichloroethane[1, 2, 5, 6]	79-00-5
cis-1,2-Dichloroethene[1, 2, 5, 6, 7]	156-59-4	Trichloroethene[1, 2, 5, 6]	79-01-6
trans-1,2-Dichloroethene[1, 2, 5, 6, 7]	156-60-5	Trichlorofluoromethane[1, 2, 5, 6, 7]	75-69-4
Dichlorofluoromethane[4]	75-43-4	1,2,3-Trichloropropane[1, 2, 6, 7]	96-18-4
1,2-Dichloropropane[1, 2, 5, 6, 7]	78-87-5	Trichlorotrifluoroethane[5, 6, 7]	76-13-1
1,3-Dichloropropane[1, 7]	142-28-9	1,2,4-Trimethylbenzene[1, 9]	95-63-6
2,2-Dichloropropane[1, 7]	590-20-7	1,3,5-Trimethylbenzene[1, 9]	108-67-8
1,1-Dichloropropene[1, 7]	563-58-6	Vinyl acetate[2, 6, 7]	108-05-4
cis-1,3-Dichloropropene[1, 2, 5, 6, 7]	10061-01-5	Vinyl chloride[1, 2, 5, 6, 7]	75-01-4
trans-1,3-Dichloropropene[1, 2, 5, 6, 7]	10061-02-6	o-Xylene[9]	95-47-6
2,3-Dichloro-1-propene[4]	78-88-6	m-Xylene[9]	108-38-3
Diethyl ether[4]	60-29-7	p-Xylene[9]	106-42-3
3,3-Dimethyl-1-butanol[3]	624-95-3	Xylene (total)[1, 2, .5, 6, 7, 8, 9]	1330-20-7
[1] - Laboratory normal 8260 compound		[6] - Appendix I compound	
[2] - Appendix IX compound		[7] -Appendix II compound	
[3] - Oxygenate		[8] - Skinner list	
[4] - Additional compounds by request only		[9] - NY Stars List	
[5] TCL list (OLM 04.2 list)		a = Chemical Abstract Service Registry Number	

1.2 Reporting Limits: The RL for an individual compound is instrument-dependent and also dependent on the choice of sample preparation/introduction method. The RL analyte concentration is defined by the lowest non-zero standard in the calibration curve. Using standard quadrapole instrumentation and the purge-and-trap technique, RLs are approximately:

5 mL Water Purge (µg/L)	10 mL Water Purge (µg/L)	Soil/Sediment[1] (µg/kg)
1	0.5	2

Sample RLs are highly matrix-dependent. The RLs listed are provided for guidance and may not always be achievable.

[1] RLs listed for soil/sediment are based on wet weight. When data are reported on a dry weight basis, RLs are higher, based on the percent dry weight in each sample.

Typical target compound RLs are shown in Table 1. For the most current analyte RL, refer to the current Control Limits Manual and LIMS.

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Table 1. Chromatographic Retention Times and Typical Reporting Limits

Compounds	Retention Time (minutes)	Typical Reporting Limits		
		5 mL-Purge (µg/L)	10 mL-Purge (µg/L)	Soils (µg/kg)
Dichlorodifluoromethane	1.13	1	0.5	2
Chloromethane	1.25	1	0.5	2
Vinyl chloride	1.31	1	0.5	2
Bromomethane	1.51	1	0.5	2
Chloroethane	1.57	1	0.5	2
Dichlorofluoromethane	1.67	1	0.5	2
Trichlorofluoromethane	1.71	1	0.5	2
Ethanol	1.80	100	50	200

Compounds	Retention Time (minutes)	Typical Reporting Limits		
		5 mL-Purge (µg/L)	10 mL-Purge (µg/L)	Soils (µg/kg)
Diethyl ether	1.87	10	2.5	10
Acrolein	1.95	10	5.0	10
Dimethylbutane	1.98	2	NA	2
Trichlorotrifluoroethane	2.00	1	0.5	2
1,1-Dichloroethene	2.01	1	0.5	2
Acetone	2.03	50	5.0	50
Iodomethane	2.11	10	2.5	10
Bromoethane	2.11	2	NA	2
Isopropyl alcohol	2.10	20	50	20
Carbon disulfide	2.15	1	0.5	2
Acetonitrile	2.16	10	20	10
Allyl chloride	2.18	10	10	10
Pentane	2.18	2	1	2
Methyl acetate	2.22	10	10	10
Methylpentane	2.26	2	0.5	2
Methylene chloride	2.28	5	2.5	10
t-Butyl alcohol	2.34	10	10	100
Acrylonitrile	2.42	10	2.5	10
trans-1,2-Dichloroethene	2.44	1	0.5	2
MTBE	2.43	1	0.5	2
2-Nitropropane	2.53	5	5	5
Hexane	2.60	2	0.5	10
1,1-Dichloroethane	2.69	1	0.5	2
Vinyl acetate	2.71	10	5.0	10
Chloroprene	2.71	5	5	5
Diisopropyl ether	2.72	10	2.5	10
ETBE	2.94	1	0.5	10
Methylcyclopentane	2.94	2	1	4
Ethyl acetate	3.03	5	5	50
cis-1,2-Dichloroethene	3.04	1	0.5	2
2,2-Dichloropropane	3.05	1	0.5	2
2-Butanone	3.05	50	2.5	50
Propionitrile	3.05	20	10	50
Methacrylonitrile	3.15	20	20	50
Bromochloromethane	3.20	1	0.5	2
Chloroform	3.25	1	0.5	2
Tetrahydrofuran	3.23	20	10	20
t-Butyl formate	3.28	10	100	100
1,1,1-Trichloroethane	3.38	1	0.5	2
Cyclohexane	3.41	5	1	10
Carbon tetrachloride	3.48	1	0.5	2
1,1-Dichloropropene	3.48	1	0.5	2
Isobutanol	3.48	50	25	100
Benzene	3.62	1	0.5	2
1,2-Dichloroethane	3.63	1	0.5	2
t-Amyl alcohol	3.60	10	100	100
TAME	3.68	1	0.5	10
Heptane	3.73	2	2	4
n-Butanol	3.94	20	50	100

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Compounds	Retention Time (minutes)	Typical Reporting Limits		
		5 mL-Purge (µg/L)	10 mL-Purge (µg/L)	Soils (µg/kg)
Trichloroethene	4.06	1	0.5	2
Methylcyclohexane	4.20	5	0.5	10
1,2-Dichloropropane	4.22	1	0.5	2
Methyl methacrylate	4.24	5	1	10
2,3-Dichloro-1-propene	4.25	1	0.5	2
Dibromomethane	4.30	1	0.5	2
Bromodichloromethane	4.42	1	0.5	2
1,4-Dioxane	4.32	100	100	100
2-Chloroethylvinyl ether	4.63	10	2.5	10
cis-1,3-Dichloropropene	4.75	1	0.5	2
ETBA	4.76	10	0.5	10
4-Methyl-2-pentanone	4.86	10	2.5	10
Toluene	5.01	1	0.5	2
trans-1,3-Dichloropropene	5.18	1	0.5	2
Ethyl methacrylate	5.25	10	10	10
3,3-Dimethyl-1-butanol	5.29	10	10	NA
1,1,2-Trichloroethane	5.33	1	0.5	2
Tetrachloroethene	5.46	1	0.5	2
1,3-Dichloropropane	5.47	1	0.5	2
2-Hexanone	5.54	10	5.0	10
Butyl acetate	5.59	20	10	40
Dibromochloromethane	5.65	1	0.5	2
Octane	5.71	2	1	2
1,2-Dibromoethane	5.76	1	0.5	2
1-Chlorohexane	6.11	2	1	5
Chlorobenzene	6.18	1	0.5	2
1,1,1,2-Tetrachloroethane	6.18	1	0.5	2
Ethylbenzene	6.27	1	0.5	2
m & p-Xylene	6.38	1	0.5	2
o-Xylene	6.73	1	0.5	2
Styrene	6.75	1	0.5	2
Bromoform	6.91	1	0.5	2
Nonane	7.03	2	1	2
Isopropylbenzene (Cumene)	7.06	1	0.5	2
cis-1,4-Dichloro-2-butene	7.08	5	5	10
Cyclohexanone	7.10	50	50	50
Bromobenzene	7.34	1	0.5	2
1,1,2,2-Tetrachloroethane	7.34	1	0.5	2
1,2,3-Trichloropropane	7.38	1	0.5	2
trans-1,4-Dichloro-2-butene	7.40	10	1.0	10
n-Propylbenzene	7.45	1	0.5	2
2-Chlorotoluene	7.52	1	0.5	2
4-Chlorotoluene	7.62	1	0.5	2
1,3,5-Trimethylbenzene	7.61	1	0.5	2
DIBK	7.70	10	10	50
Pentachloroethane	7.88	5	5	10
tert-Butylbenzene	7.92	1	0.5	2
1,2,4-Trimethylbenzene	7.97	1	0.5	2
sec-Butylbenzene	8.13	1	0.5	2

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Compounds	Retention Time (minutes)	Typical Reporting Limits		
		5 mL-Purge (µg/L)	10 mL-Purge (µg/L)	Soils (µg/kg)
p-Isopropyltoluene	8.27	1	0.5	2
1,3-Dichlorobenzene	8.23	1	0.5	2
1,4-Dichlorobenzene	8.31	1	0.5	2
Benzyl chloride	8.41	10	5	20
1,2-Dichlorobenzene	8.67	1	0.5	2
n-Butylbenzene	8.67	1	0.5	2
1,2-Dibromo-3-chloropropane	9.43	10	0.5	10
1,3,5-Trichlorobenzene	9.50	1	0.5	2
1,2,4-Trichlorobenzene	10.36	1	0.5	2
Hexachlorobutadiene	10.58	1	0.5	2
Naphthalene	10.65	5	5	5
1,2,3-Trichlorobenzene	10.94	1	0.5	2
2-Methylnaphthalene	11.85	10	5	10
1-Methylnaphthalene	12.12	10	5	10
INTERNAL STANDARDS/SURROGATES				
Fluorobenzene	3.75			
Chlorobenzene-d_5	6.10			
1,4-Dichlorobenzene-d_4	8.24			
1,2-Dichloroethane-d_4	3.53			
Dibromofluoromethane	3.30			
Toluene-d_8	4.91			
4-Bromofluorobenzene	7.16			

1.3 There are various techniques by which these compounds may be introduced into the GC/MS system. Purge-and-trap, by Methods 5030 /NV05-107 (aqueous samples) and 5035 / NV05-108 (solid and waste oil samples), is used for volatile organic analytes.

1.4 If for any reason a part of this method cannot be followed, seek the guidance of the Department Supervisor or the Laboratory Technical Director. All abnormalities must be noted on the data or the benchsheet and in the Laboratory Information Management System (LIMS).

2.0 Summary of Method

2.1 The volatile compounds are introduced into the gas chromatograph by the purge-and-trap method. The analytes are introduced directly to a capillary column for analysis. The column is temperature-programmed to separate the analytes, which are then detected with a mass spectrometer (MS) directly interfaced to the gas chromatograph (GC).

2.2 Identification of target analytes is accomplished by comparing their mass spectra with the electron impact spectra of authentic standards. Quantitation is accomplished by comparing the response of a major ion relative to an internal standard using at least a five-point calibration curve.

3.0 Definitions

See TestAmerica Nashville's Quality Assurance Manual Appendix 5 for laboratory definitions. Also, refer to Controlled Document QAF-45, TestAmerica Nashville Acronyms, Keywords, and Definitions.

4.0 Interferences

4.1 Major contaminant sources are volatile materials in the laboratory and impurities in the inert purging gas and in the sorbent trap. The use of non-polytetrafluoroethylene (PTFE) thread sealants, plastic tubing, or flow controllers with rubber components are avoided, since such

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materials out-gas organic compounds which are concentrated in the trap during the purge operation. Analyses of blanks provide information about the presence of contaminants. When potential interfering peaks are noted in blanks, perform maintenance. **Subtracting blank values from sample results is not permitted.**

4.2 Contamination may occur when a sample containing low concentrations of volatile organic compounds is analyzed immediately after a sample containing high concentrations of volatile organic compounds. A technique to prevent this problem is to rinse the purging apparatus and sample syringes with two portions of organic-free reagent water between samples.

4.3 Special precautions are taken to analyze for methylene chloride. The analytical and sample storage areas are isolated from all atmospheric sources of methylene chloride. Otherwise, random background levels result. Since methylene chloride permeates through PTFE tubing, all gas chromatography carrier gas lines and purge gas plumbing is constructed from stainless steel or copper tubing.

4.4 Samples can be contaminated by diffusion of volatile organics (particularly methylene chloride and fluorocarbons) through the septum seal of the sample container into the sample during shipment and storage. A trip blank, prepared from organic-free reagent water and carried through the sampling, handling, and storage, serve as a check on such contamination.

5.0 Safety

Employees must abide by the policies and procedures in the Corporate Safety Manual and this document. This procedure may involve hazardous material, operations and equipment. This method does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats, and closed-toe, nonabsorbent shoes are a minimum.

5.1 Specific Safety Concerns or Requirements:

- The gas chromatograph and mass spectrometer contain zones that have elevated temperatures. The analyst needs to be aware of the locations of those zones, and should cool them to room temperature prior to working on them.
- The mass spectrometer is under vacuum. The mass spectrometer must be brought to atmospheric pressure prior to working on the source.
- There are areas of high voltage in both the gas chromatograph and the mass spectrometer. Depending on the type of work involved, either turn the power to the instrument off, or disconnect it from its source of power.

5.2 Primary Materials Used: The following is a list of the materials used in this method, which have a serious or significant hazard rating. **Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

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Material[1]	Hazards	Exposure Limit[2]	Signs and symptoms of exposure
Methanol	Flammable Poison Irritant	200 ppm-TWA	A slight irritant to the mucous membranes. Toxic effects exerted upon nervous system, particularly the optic nerve. Symptoms of overexposure may include headache, drowsiness and dizziness. Methyl alcohol is a defatting agent and may cause skin to become dry and cracked. Skin absorption can occur; symptoms may parallel inhalation exposure. Irritant to the eyes.
Hydro-chloric Acid	Corrosive Poison	5 ppm-Ceiling	Inhalation of vapors causes coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and cause damage to the eyes. Contact causes severe burns and permanent eye damage.
Sodium bisulfate	Irritant	None	Causes mild to severe irritation to the eyes. Prolonged exposure causes burn if not flushed with water. Causes mild irritation to skin. Prolonged exposure causes burn if not flushed with water.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

6.0 Equipment and Supplies

6.1 Instrumentation

- Purge-and-trap device for aqueous samples at ambient temperature, described in Method 5030 / NV05-107.
- Purge-and-trap device for solid samples at 40°C, described in Method 5035 / NV05-108.
- Gas chromatography/mass spectrometer/data system
  - Gas chromatograph (HP): Analytical system complete with a temperature-programmable gas chromatograph suitable for splitless injection with appropriate interface for sample introduction device. The system includes all required accessories, including syringes, analytical columns, and gases.

Injector temperature:	250°C
MS interface temperature:	260°C
Carrier gas (He) flow rate:	Constant flow of 1.0 mL/minute.
Initial temperature:	45°C hold for 6 minutes.
Temperature program:	13°C/minute to 150°C; 18°C/minute to 220°C
Final temperature:	220°C, hold until all expected compounds have eluted (2 minutes)
Split ratio (min.)	1:10

May vary by instrument; see maintenance log for current program.

- The capillary column is directly coupled to the source.
- Gas chromatographic column: DB-624, 20 m x 0.18 mm with 1.0 µm film thickness.
- Mass spectrometer: Capable of scanning from 35 to 300 amu every 0.6 - 2 seconds using 70 volts (nominal) electron energy in the electron impact ionization mode. To ensure sufficient precision of mass spectral data, the desirable MS scan rate allows acquisition of at least five spectra while a sample component elutes from the GC.
- Data system (HP Chem Station with Enviroquant): A computer system that allows the continuous acquisition and storage on machine-readable media of all mass spectra obtained throughout the duration of the chromatographic program. The computer has software that allows searching the GC/MS data file for ions of a specified mass and

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plotting such ion abundances versus time or scan number. This type of plot is defined as an Extracted Ion Current Profile (EICP). Software is used that allows integrating the abundances in any EICP between specified time or scan-number limits. The most recent version of the EPA/NIST Mass Spectral Library is also available.

6.2 Supplies

- Microsyringes, 10, 25, 100, 250, 500, and 1,000 µL.
- Syringes, 5, 10, or 25 mL.
- Balance, analytical, capable of weighing 0.0001 g, and top-loading, capable of weighing 0.1 g.
- Glass scintillation vials, 20 mL, with PTFE-lined screw-caps or glass culture tubes with PTFE-lined screw-caps.
- Disposable pipets, Pasteur.
- Volumetric flasks, Class A, 10 mL, 50 mL and 100 mL, with ground-glass stoppers.
- Spatula, stainless steel, or wooden tongue depressor.
- Helium for carrier gas.
- Nitrogen for purge-and-trap gas.
- Narrow-range pH paper.
- Residual chlorine test strips.

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7.0 Reagents and Standards

7.1 Reagent grade chemicals are used in all tests. Unless otherwise, indicated, it is intended that all reagents conform to the specifications of the Committee on Analytical Reagents of the Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination. See the QA Manual and SOP Reagent and Standard Purchase / NV08-214 for more information on reagent chemicals, such as shelf-life and storage.

7.2 Reagent water, analyte-free, ASTM Type II or equivalent.

7.3 Methanol, CH_3OH: Purge-and-trap grade or equivalent, demonstrated to be free of analytes at the MDL. Store apart from other solvents.

7.4 Hydrochloric acid (1:1 v/v), HCl: Carefully add a measured volume of concentrated HCl to an equal volume of organic-free reagent water.

7.5 Stock solutions: Stock solutions are prepared from pure standard materials or purchased as certified solutions. Prepare stock standard solutions in methanol, using assayed liquids. Any specific standards or procedure for making standards mentioned in this SOP may be substituted with equivalent standards or procedures. See standard log for specific standard information.

7.5.1 **Primary Standards for Calibration**

		For Working Standard		
Name of standard	**Vendor[2]/Conc (µg/mL)**	**Volume used (mL)**	**Final Volume (mL)**	**Conc. (µg/mL)**
Custom 8260 VOC mega-mix[1] without gases	Restek 550174/2000-10000	2.5	50	100 – 500
Ketones	Restek 56664/10000	2.5	50	500
Acrolein/Acrylonitrile	Supelco M-603-10X /10000	2.5	50	500
Vinyl acetate	Restek 57488/10000	2.5	50	500
2-Chloroethylvinyl ether	Restek 57489 /10000	2.5	50	500
Miscellaneous Mix	Restek 56451/2000-200,000	2.5	50	100-10,000
1-Methylnaphthalene	Restek 31283/1000	0.1	2	50

		For Working Standard		
Name of standard	Vendor[2]/Conc (µg/mL)	Volume used (mL)	Final Volume (mL)	Conc. (µg/mL)
Piano paraffins	Supelco 44585-U/1000	0.5	10	50
Gas Mix	Restek 560385/100	Combined with the above mixture or added separately to the QC sample to achieve the desired concentration as described in the Calibration section.		
Appendix IX Mix	Restek 550020/100-1000	See 7.5.1.3.2		

1 Custom 8260 VOC mix has variable concentrations. See the standard log for exact compound concentrations.
2 The vendors/catalog numbers are recommended, equivalent products are acceptable.

7.5.1.1 Transfer the stock standard solution into a bottle with a PTFE-lined screw-cap. Store, with minimal headspace and protected from light, at -10°C or less or as recommended by the standard manufacturer. Return standards to the freezer as soon as the analyst has completed mixing or diluting the standards to prevent the evaporation of target compounds.

7.5.1.2 **Frequency of Standard Preparation**

7.5.1.2.1 Monitor standards for the permanent gases frequently by comparison to the initial calibration curve. Prepare fresh standards if this check exceeds a 20% drift. **Standards for gases usually need to be replaced after one week or as recommended by the standard manufacturer, unless the acceptability of the standard can be documented.** Dichlorodifluoromethane and dichloromethane are usually the first compounds to evaporate from the standard and, therefore, are to be monitored very closely when standards are held beyond one week.

7.5.1.2.2 Monitor standards for the non-gases frequently by comparison to the initial calibration, i. e., CCV. Prepare fresh standards if this check exceeds a 20% drift. Undiluted standards for non-gases usually need to be replaced after **six months** or as recommended by the standard manufacturer, unless the acceptability of the standard can be documented. Standards of reactive compounds such as 2-chloroethyl vinyl ether and styrene may need to be prepared more frequently.

7.5.1.3 **Secondary dilution standards:** Using stock standard solutions, prepare secondary dilution standards in methanol containing the compounds of interest, either singly or mixed together. Secondary dilution standards are stored with minimal headspace and, except for gases, are good for one month. Store in a vial with minimal headspace. Replace secondary standards for gases after one week unless the acceptability of the standard can be documented. When using premixed certified solutions, store according to the manufacturer's documented holding time and storage temperature recommendations. Handle and store standards as stated above and return them to the freezer as soon as standard mixing or diluting is completed to prevent the evaporation of volatile target compounds.

7.5.1.3.1 The working calibration standard for the Non-Gas mixture is made by adding 2.5 mL of each of the first six standards in the Primary Standard table above in 50.0 mL methanol in a Class A volumetric. The Gas Standard is added right before use as described in the calibration section.

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7.5.1.3.2 The Appendix IX working standard is made separately: Make a 1:1 standard of the Appendix IX Mix with methanol in a Class A volumetric for a 50-500 µg/L standard.

7.5.2 **Internal Standard/Surrogate Standard Mix (IS/SS)**

7.5.2.1 The internal standards are fluorobenzene, chlorobenzene-d_5, and 1,4-dichlorobenzene-d_4. Prepare internal standard stock and secondary dilution standards in methanol. Stock standard is 2500 µg/mL, Ultra CUS-7787, or equivalent.

7.5.2.2 The surrogates are toluene-d_8, 4-bromofluorobenzene (the GC/MS tuning standard), 1, 2-dichloroethane-d_4, and dibromofluoromethane. Stock standard is 2500 µg/mL, Ultra CUS-5660, or equivalent.

7.5.2.3 Prepare a 250 µg/mL IS/SS standard by diluting 5.0 mL of stock internal standard (2500 µg/mL) and 5.0 mL stock surrogate standard (2500 µg/mL) to a final volume of 50.0 mL of methanol in a Class A volumetric.

7.5.3 **Second-Source Standards for Laboratory Control Sample (LCS) and Matrix Spike (MS) Standards:** The MS/MSD and LCS are all spiked with a **second-source** Spike Standard that contains all the reported 8260 compounds for the project.

To prepare the **2nd Source Spike Standard**:

1. First prepare a 2nd Source Non-Gas Standard using the volumes indicated:

2nd Source Non-Gas Standards

		For 2nd Source Non-Gas Spiking Standard		
Name of Standard	**Vendor/Conc. (µg/mL)**	**Volume used (mL)**	**Final Volume (mL)**	**Conc (µg/mL)**
Custom 8260 mega-mix [1]without gases	Ultra DWM- 589N/2000 - 10000	2.5	50	100-500
Keton Mix	Ultra CUS-6783/10000	2.5	50	500
Acrolein/ Acrylonitrile	Ultra AMN-803/10000	2.5	50	500
Vinyl acetate	Ultra CUS-6784/10000	2.5	50	500
2-Chloroethylvinyl ether	Ultra EPA-1016/10000	2.5	50	500
Miscellaneous Mix	Ultra CUS-7854/2000 – 200,000	2.5	50	100-10,000
1-Methylnaphthalene	Ultra EPA-1225/1000	0.1	2	50

1 Custom 8260 VOC mix has variable concentrations. See the standard log for exact compound concentrations.
2 The vendors/catalog numbers are recommended, equivalent products are acceptable.

2. Second, make a 1:1 mixture of the above mixes with the Gas Mix, Ultra CUS-5661, or equivalent (100 µg/mL each compound).

7.5 **Sea or Ottawa sand** for blank and LCS soil matrix.

7.6 **Sodium Bisulfate** for soil sample preservation. See SOP 5035 / NV05-108.

8.0 Sample Collection, Preservation, Shipment and Storage

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Matrix	Sample Container	Min. Sample Size	Preservation	Holding Time From Collection to Analysis	Reference
Water	3 x 40-mL VOAs	40 mL	pH < 2 with hydrochloric acid. Cool 0-6°C, No headspace. Keep in dark.	14 days	SW846 Section 2.0
Low-concentration Soil	2 pre-weighed vials, stirring bar	5 g	0-6°C, 5 mL preservative[1]		
	2 EnCores™		0-6°C, Add 5 g sample and 5 mL preservative to pre-weighed vial with stirring bar within 48 hours of collection		
High concentration Soil	2-oz. glass or 25 g Encore™	5g or 25 g	0-6°C, Add 1 mL methanol/gram soil		

[1]0.2 gram sodium bisulfate/mL reagent water
See SOPs 5030 / NV05-107 for waters and 5035 / NV05-108 for soils/solids, including the soil freezing option, with or without water.

Analysis Method	Sample Storage	Holding Times from Date and Time of Collection			
		MeOH Addition	Shipping	Extraction	Analysis
Wisconsin VOC Soils	VOC vial	Immediately	4 days	21 days	21 days
	Brass Tube	within 2 hours	4 days	21 days	21 days
	EnCore™	within 48 hours	40 hours	21 days	21 days

9.0 Quality Control

The laboratory maintains a formal quality assurance program and records to document the quality of the data generated.

9.1 Sample QC: The following QC is run **every batch of no more than 20 samples, except samples from OK and WY where a batch is no more than 10 samples:**

QC Check	Frequency	Acceptance Criteria[1]	Corrective Action[2]
Method blank	One per analytical prep batch and after calibration (see Section 9.2)	No analytes detected ≥ ½ RL or MDL, whichever is greater	Correct problem then re-prep[3] and analyze method blank and all samples processed with the contaminated blank. If target > 10x blank, report but qualify.
LCS[6] for all analytes must be from a 2nd source.	One[6] per prep batch	See Control Limits Manual[4,7]	Re-prep[3] and analyze the LCS and all samples in the affected analytical batch. If high and samples are ND, report. If low, re-prep.
MS/MSD must be from a 2nd source.	One per batch per matrix, if insufficient sample for MS/MSD, then analyze a LCS.	See Control Limits Manual	None (LCS is used to determine if data is acceptable).
Surrogate	Every sample, spike, standard, and blank.	See Control Limits Manual[5]	Check system, re-analyze, re-prep[3], may qualify. If %recovery is high and the sample is ND, it is acceptable to report. If low, re-prep and rerun.
pH check	All water samples.	pH ≤2.	If the pH is > 2, then comment the data and LIMS.
Residual chlorine	Each sample.	Residual chlorine must be	If the residual chlorine is positive,

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QC Check	Frequency	Acceptance Criteria[1]	Corrective Action[2]
check (North Carolina samples only)		negative.	then comment the data, and LIMS.

[1]This is a summary of the acceptance criteria.
[2]All abnormalities must be noted on the data, the benchsheet, and in LIMS.
[3]If unable to re-prep the samples because of insufficient sample volume or holding time has expired, then place a comment on the benchsheet and in LIMS.
[4]If the LCS exceeds the upper control limit AND a sample from that batch is greater than the RL, re-prep and re-analyze the batch. If the LCS exceeds the upper control limit AND the sample from that batch is less than the RL, the data is acceptable to report.
[5]If the surrogate % recovery exceeds the upper control limit AND a sample is greater than the RL, re-prepare and re-analyze the sample. If the surrogate % recovery exceeds the upper control limit AND the sample is less than the RL, data is acceptable to report. If the surrogate % recovery is lower than the lower control limit, re-prepare the sample. OH VAP requires all surrogates to be in control; otherwise, the samples must be re-prepared and re-analyzed.
[6]All AZ, TX, and WV samples require a LCS duplicate in each batch.
[7]See Attachment 3 for South Carolina LCS acceptance criteria.

- A **Method Blank** is run with each analytical batch to ensure that the total system (introduction device, transfer lines and GC/MS system) is free of contaminants. Each time a set of samples is analyzed or there is a change in reagents, a method blank is analyzed as a safeguard against chronic lab contamination. The blank is carried through all stages of the sample preparation and measurement using the appropriate blank matrix (reagent water or sand).
  - If the method blank is detected for an analyte ≥ ½ RL or MDL, whichever is greater, it must be reanalyzed along with all samples processed against the contaminated blank. However, if the associated samples are non-detect, then reanalysis of the Method Blank is not required.
- A **Laboratory Control Sample (LCS)** is included with each analytical batch. The LCS consists of an aliquot of a clean (control) matrix (reagent water or sand) similar to the sample matrix and of the same weight or volume. The LCS is spiked with the same analytes from a **second** source. When the results of the matrix spike analysis indicate a potential problem due to the sample matrix itself, the LCS results are used to verify that the laboratory can perform the analysis in a clean matrix.
  - If the LCS fails, reanalyze the LCS and all samples in the affected analytical batch. If the LCS recovery is high, and the associated samples are ≤ RL, corrective action is not necessary, but data must be flagged.

Matrix	LCS Preparation	Final Concentration
Water	Add 50 µL of the 2nd Source Spike Standard to 50.0 mL reagent water in a Class A volumetric flask.	50 – 5000 µg/L
Low-concentration Soil	Add 5 µL of the 2nd Source Spike Standard to a VOA vial containing 5.0 g sand and 5 mL preservative and a stirring bar.	50 - 5000 µg/kg
High-concentration Soil (analyzed as waters)	Add 50 µL of the 2nd Source Spike Standard to 50.0 mL reagent water in a Class A volumetric flask.	50 - 5000 µg/kg

- **Matrix Spike/Matrix Spike Duplicate:** Documenting the effect of the matrix includes the analysis of at least one matrix spike/matrix spike duplicate pair for each batch.

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Matrix	MS/MSD Preparation	Final Concentration
Water Batch	Add 45 µL of the 2nd Source Spike Standard to the client's sample in VOA vials.	50 – 5000 µg/L
Low-concentration Soil Batch	Add 5 µL of the 2nd Source Spike Standard to a VOA vial containing 5 g preserved client sample (with stirring bars).	50 - 5000 µg/kg
High-concentration Soil Batch (analyzed as waters)	Add 1.0 mL of the methanol-extract-of-client-sample and 50 µL of the 2nd Source Spike Standard and dilute with reagent water in a 50-mL, Class A volumetric.	50 - 5000 µg/kg

- **Surrogate standards**: The analyst monitors both the performance of the analytical system and the effectiveness of the method in dealing with each sample matrix by spiking each sample, QA/QC standard, and blank with surrogate compounds which are not expected to be affected by method interferences. The surrogate and internal standards are prepared together as described in Section 7.
  - The IS/SS standard mix (250 µg/mL each) is added by the autosampler (1 µL) during all analyses with the exception of the calibration.

Purge Volume, mL	Concentration of IS/SS Standards in Sample, µg/L
5	50
10	25

- **pH Check:** The analyst must document that each sample has a pH ≤ 2 or not by checking with narrow-range pH paper. The pH check is performed after sample analysis to avoid contamination and creation of a headspace in the sample vials. Record as pH <2 or >2.
- **Residual Chlorine Check:** The analyst must document the presence/absence of residual chlorine in North Carolina samples by checking with residual chlorine test strips.

9.2 Instrument QC

QC Check	Frequency	Acceptance Criteria[1]	Corrective Action[2]
Check of mass spectral ion intensities, i. e., BFB Tune	Prior to initial calibration or Continuing calibration verification, every 12 hours	Refer to criteria for Tune criteria	Retune the instrument and verify (instrument maintenance may be needed).
Minimal five-point initial calibration for all target analytes. Single-point surrogate calibration.	Initial calibration prior to sample analysis. Perform instrument re-calibration once per year minimum.	SPCCs average RF ≥ 0.30 or 0.1 depending on the compound and %RSD for RFs for CCCs ≤ 30% and all other target analytes %RSD for RF ≤ 15%. $r^2 \geq 0.99$ or $r \geq 0.995$	Correct problem then repeat initial calibration.
Initial calibration verification (ICV), must be from a 2nd source.	Immediately following each initial calibration.	All analytes within 30% of expected value.	Correct problem then repeat initial calibration. ICV must be run prior to reporting samples.
Continuing Calibration Verification (CCV).	Daily, before sample analysis and every 12 hours of analysis time.	CCCs: ≤20% difference (when using RFs) or drift (when using least squares regression). **All other target compounds ≤ 20%, except for specific compounds which may have a % difference ≤ 40%.**	Correct problem then repeat CCV (re-calibrate if necessary) and re-analyze any samples processed with that CCV. If the CCV is high and the sample is ND, it is acceptable to report.[3]

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QC Check	Frequency	Acceptance Criteria[1]	Corrective Action[2]
Continuing Calibration Blank.	After each CCV.	< ½ RL or MDL, whichever is greater.	Correct problem, repeat.
Internal Standards[3]	Every sample, standard and blank.	Retention time ±30 seconds from retention time of the mid-point std. in the ICAL. EICP area within -50% to +100% of ICAL mid-point std.	Inspect mass spectrometer and GC for malfunctions; mandatory re-analysis of samples analyzed while system was malfunctioning.
Retention time window calculated for each analyte	Each sample.	Relative retention time (RRT) of the analyte within 0.06 RRT units of the RRT of the internal standard.	Correct problem then re-analyze all samples analyzed since the last retention time check.
MDL verification	Minimum yearly.	Detectible.	Re-evaluate MDL standard used and MDL; see Technical Director.

[1] This is a summary of the acceptance criteria.
[2] All abnormalities must be noted on the data, the benchsheet, and in LIMS.
[3]Target compounds associated with failed internal standards must be re-analyzed (undiluted if possible) if additional sample is available; if not available, qualify data in LIMS.

- **BFB Tuning**: At the beginning of each 12-hour analytical shift and **prior** to the analysis of samples or calibration standards, inject 50 ng or less of the 4-bromofluorobenzene standard into the GC/MS system (1 µL of 250 µg/mL standard /50 mL reagent water, purged at a 1:10 split for a 25 ng on the column). (BFB is one of the surrogate compounds.) The resultant mass spectra for the BFB **must** meet the tuning criteria below before sample analysis begins.

BFB (4-Bromofluorobenzene) Mass Intensity Criteria

m/z	Required Intensity (relative abundance)
50	15 to 40% of m/z 95
75	30 to 60% of m/z 95
95	Base peak, 100% relative abundance
96	5 to 9% of m/z 95
173	Less than 2% of m/z 174
174	Greater than 50% of m/z 95
175	5 to 9% of m/z 174
176	Greater than 95% but less than 101 % of m/z 174
177	5 to 9% of m/z 176

- Three options are available for acquiring the spectra for reference to meet the BFB tuning requirements:

Option 1 It is recommended that each initial tune verification utilize the "Autofind" function and be set up to look at the apex ± 1 scan and average the three scans. Background correction is required prior to the start of the peak but no more than 20 scans before. Background correction **cannot** include any part of the target peak. Sometimes the instrument does not always correctly identify the apex on some peaks when the peak is not perfectly shaped. It is acceptable to manually identify and average the apex peak ± 1 scan and background correct.

Option 2 The scan across the peak at one half peak height may be averaged and background-corrected.

Option 3 A single scan at the apex (only) may also be used for the evaluation of the tune. Background correction is still required.

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Note: It is acceptable to adjust parameters within the specifications set by the manufacturer or the analytical method to properly tune the instrument. If the tune verification does not pass it may be necessary to clean the source or perform additional maintenance. Document any maintenance in the instrument log. **Excessive adjusting (more than two tries) without clear documentation is not allowed. No more than two consecutive tunes may be attempted. Perform necessary maintenance.**

- Note: All subsequent standards, samples, controls, and blanks associated with a BFB tune **must** use identical mass spectrometer instrument conditions.

- **Calibration standards**: See Section 10.2.
  - **Initial System Performance Check Compounds (SPCCs):** A system performance check is made before the calibration curve is used. Five compounds (the System Performance Check Compounds) are checked for a minimum average response factor, compound instability, and degradation caused by contaminated lines or active sites in the system. These compounds are chloromethane, 1,1-dichloroethane, bromoform, chlorobenzene, and 1,1,2,2-tetrachloroethane. The minimum mean response factors for the volatile SPCCs must be met and are as follows:

Chloromethane	0.10
Bromoform	0.10
1,1,2,2-Tetrachloroethane	0.30
1,1-Dichloroethane	0.10
Chlorobenzene	0.30

Example problems include:
- Chloromethane is the most likely compound to be lost if the purge flow is too fast.
- Bromoform is one of the compounds most likely to be purged very poorly if the purge flow is too slow. Cold spots and/or active sites in the transfer lines may adversely affect response. Response of the quantitation ion (m/z 173) is directly affected by the tuning of BFB at ions m/z 174/176. Increasing the m/z 174/176 ratio relative to m/z 95 may improve bromoform response.
- Tetrachloroethane and 1,1-dichloroethane are degraded by contaminated transfer lines or active sites in trapping materials.

  - **Initial Calibration check compounds (CCCs):** The purpose of the CCCs is to evaluate the calibration from the standpoint of the integrity of the system. High variability for these compounds may be indicative of system leaks or reactive sites in the system. Meeting the CCC criteria is **not** a substitute for successful calibration of the target analytes. The CCCs are:

1,1-Dichloroethene	Toluene
Chloroform	Ethylbenzene
1,2-Dichloropropane	Vinyl chloride

- Calculate the standard deviation (SD) and relative standard deviation (RSD) of the response factors for **all** target analytes from the initial calibration with the equations in Section 11.
- The RSD must be less than or equal to **15% for each target analyte**; however, the RSD for each individual Calibration Check Compound (CCC) must be equal or less

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than 30%. If an RSD of greater than 30% is measured for any CCC, then corrective action to eliminate a system leak or contamination and/or column reactive sites is necessary before re-attempting calibration. The CCCs may not be in the project target list. If that is the case, each target must have a RSD < 15% or a correlation coefficient $r \geq 0.995$ ($r^2 \geq 0.99$) as calculated by the equations in Section 11.

- **Initial Calibration Verification (ICV)** is verified immediately after calibration using the introduction technique used for samples. Analyze a calibration standard at a concentration near the midpoint concentration for the calibrating range of the GC/MS
  - The ICV is made from the **second-source** standards, one at a time, as needed, to be run after an initial calibration: Add 2.5 mL of each of the six standards in the Second-source Non-Gas Standard table in Section 7.5.3 to 50 mL methanol in a Class A volumetric. Add 25 µL of this mixture and 25 µL of the Gas Mix and dilute to 50.0 mL with reagent water in a Class A volumetric for a final concentration of 50-5000 µg/L.
  - The ICV of each target must be within 30% of the expected value, with the exception of the following poor purge efficiency analytes that may be within 40% of the expected value:

Acrolein	Ethanol	2-Methylnaphthalene
t-Amyl alcohol (TAA)	t-Butyl formate (TBF)	Vinyl acetate
t-Butyl alcohol (TBA)	1-Methylnaphthalene	

  - If ICV criterion is not met, correct the problem and re-calibrate.
- **Continuing Calibration Verification (CCV):** Run every 12 hours of sample analysis, CCVs are often made each day for several instruments in the following proportions, always from the **primary** calibration standards:
  - Add 500 µL of the working calibration Non-Gas Standard and 500 µL of the primary Gas Mix to a one-liter, Class A volumetric and dilute to the mark with reagent water. The final concentration is 50-5000 µg/L. The Appendix IX CCV is the same as the Appendix IX working standard.
  - Area counts of the internal standards must be between 50 - 100% of the areas of the internal standards in the mid-point calibration standard. If not, inspect the GCMS for possible maintenance issues and then re-analyze. Contact the department supervisor for assistance in determining the appropriate course of action. **Do not report data from a failing internal standard associated with target compounds.**
  - **Continuing System Performance Check Compounds (SPCCs)**
    - A System Performance Check **must** be made during every 12-hour analytical shift. Each SPCC compound in the calibration verification standard **must** meet its minimum response factor. This is the same check that is applied during the initial calibration.
    - If the minimum response factors are not met, the system **must** be evaluated, and corrective action **must** be taken before sample analysis begins. Possible problems include standard mixture degradation, injection port inlet contamination, contamination at the front end of the analytical column, and active sites in the column or chromatographic system.
  - **Continuing Calibration Check Compounds (CCCs)**
    - After the system performance check is met, the CCCs are used to check the validity of the initial calibration, if present in the target list. Use percent difference when performing the average response factor model calibration. Use percent drift when calibrating using a regression fit model. See Section 11 for equations.
    - If the percent difference or drift for each CCC is less than or equal to 20%, the initial calibration is assumed to be valid. If the criterion is not met (i. e., greater than 20% difference or drift), for any one CCC, then corrective action **must** be taken prior to the analysis of samples. **If the CCCs are not included in the list of analytes for a**

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project and therefore not processed in the calibration standards, then all analytes must meet the 20% difference or drift criterion.

- Problems similar to those listed under SPCCs could affect the CCCs. If the problem cannot be corrected by other measures, a new five-point initial calibration must be generated. The CCC or target criteria **must** be met before sample analysis begins.

- **Continuing Evaluation of Non CCC/SPCC compounds** – The percent difference or drift for each of the non-CCC analytes is less than or equal to 20%. Recovery for some compounds with poor purge efficiency may exceed this 20% requirement and still be deemed acceptable provided all of the following criteria are met:
  - Poor performing analytes are one of the following: Acrolein, tert-Amyl alcohol (TAA), tert-Butyl alcohol (TBA), tert-Butyl formate (TBF), Ethanol, 2-Methylnaphthalene, Vinyl acetate.
  - The percent difference or drift is less than or equal to 40%.

- **Continuing Calibration Blank (CCB):** The CCB is run immediately after each CCV. If the result is not less than MDL or ½ RL, whichever is greater, correct the problem and rerun.
- **Internal Standards** are used to evaluate the effect of the sample matrix. Any samples that do not meet the internal standard criteria must be evaluated for validity. If the change in sensitivity is a matrix effect, the sample is re-analyzed to confirm. If the change in sensitivity is due to instrumental problems, all affected samples must be re-analyzed after the problem is corrected.
  - The retention times of the internal standards in the calibration verification standard are evaluated immediately after or during data acquisition. If the retention time for any internal standard changes by more than 30 seconds from that in the mid-point standard level of the most recent initial calibration sequence, then the chromatographic system must be inspected for malfunctions and corrections must be made. When corrections are made, reanalysis of samples analyzed while the system was malfunctioning is required. Note any maintenance in the logbook.
  - Internal standards permit most of the components of interest in a chromatogram to have retention times of 0.80 - 1.20, relative to one of the internal standards. Use the base peak ion from the specific internal standard as the primary ion for quantitation (see Attachment 1). If interferences are noted, use the next most intense ion as the quantitation ion.
  - **Internal standard response** - If the EICP area for any of the internal standards in the calibration verification standard and samples changes by a factor of two (-50% to +100%) from that in the mid-point standard level of the most recent initial calibration sequence, the mass spectrometer must be inspected for malfunctions and corrections must be made. When corrections are made, reanalysis of samples analyzed while the system was malfunctioning is required. Note any maintenance in the logbook.
  - The laboratory re-analyzes any sample where the internal standard fails and there is no evidence of matrix interference. If there is no matrix interference, the sample must be reanalyzed at the original dilution.
    - If the internal standard is within criteria, report the second analysis.
    - If the internal standard is still outside of criteria, the sample must be analyzed at a second dilution.
    - If the internal standard still does not meet criteria, the sample must be diluted until the internal standard meets criteria. Multiple runs may be required.
  - See Attachment 2 for the analytes corresponding to each internal standard.
- **Retention time windows**: Target analytes are identified on the basis of retention time windows.
  - Before establishing retention time windows, make sure that the chromatographic system is functioning reliably and that the operating parameters have been optimized for the

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target analytes and surrogates in the sample matrix to be analyzed.
- Establish the retention time windows for target analytes.
- The relative retention times of each target analyte in each calibration standard must agree within 0.06 relative retention time units. Late-eluting compounds usually have much better agreement.

- **Method Detection Limit Verification (MDLV):** Annually verify that the MDL is detectible; if not, re-evaluate the MDL.

10.0 Procedure

10.1 Sample Preparation

Matrix	Sample Size
Water	VOA vial
Low-concentration Soil	5 grams
High-concentration Soil	1 mL methanol extract of soil

- All samples and standard solutions are allowed to warm to ambient temperature before analysis.
- Refer to SOP 5030 / NV05-107 for waters and 5035 / NV05-108 for soils/solids.
- **Compositing aqueous samples prior to GC/MS analysis is not allowed.**
- For Wisconsin VOC soils, the following procedure must be performed for Methanol extraction of Soil/Sediment:

1	Hand-shake the sample in its vial containing Methanol vigorously for 2 minutes. Sonicate for 20 minutes.
2	Allow sediment to settle until a layer of Methanol is apparent.
3	Withdraw an appropriate aliquot of the Methanol extract for sparging and add to a VOA vial.
4	Analyze all reagent blanks and QC samples on the same instrument as that used for the samples.
5	If the responses exceed the calibration or linear range of the systems, use a smaller aliquot of Methanol extract or dilute the aqueous sample.

10.2 Initial Calibration: Refer to SOP Selection of Calibration Points / CA-T-P-002. See Section 11 for equations. Calculations are performed by vendor software and LIMS.

1	Evaluate the **BFB tuning criteria.**
2	Prepare the **Initial calibration standards** at a minimum of five different concentrations from the secondary dilution of stock standards or from a premixed certified solution in organic-free reagent water. **At least one of the calibration standards corresponds to a concentration at or below the laboratory reporting limit.** This low standard must have valid ion abundances for all monitored ions. The remaining standards define the working range of the system. Initial calibration standards are mixed from fresh stock standards and dilution standards when generating an initial calibration curve.

Initial Calibration (5-point)

Primary Working Standard or Appendix IX Working Standard (µL)	Final Volume (mL)	Concentration (µg/L)
2	100	1 - 5
4	100	2 - 10
20	100	10 - 50

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40	100	20 - 100
100	100	50 - 250
200	100	100 - 500
400	100	200 - 1000

Primary Working Standard: See Section 7.5.1.3.1 for make-up of Non-Gas Standard. Make a 1:1 mixture of the Non-Gas Standard and the Gas Standard to create the Primary Working Standard.
Appendix IX Working Standard: See Section 7.5.1.3.2 for make-up.
For 1-Methylnaphthalene, prepare the following standard concentrations: 10, 20, 50, 100, 200 µg/mL.
For the paraffin group, prepare the following standard concentrations: 2, 5, 10, 20, 50, 100, 200 µg/mL.
1 µL of IS/SS Standard at 250 µg/mL is added by the autosampler to 5 mL for a 50-µg/L concentration in the standards and samples with a 5-mL purge. See Section 9.1 for make-up and final concentrations for other purge volumes. The surrogate calibration is a single-point.

- All target analytes for a particular analysis must be included in the initial calibration and calibration verification standard(s). These target analytes may not include the entire list of analytes for which the method has been demonstrated. **However, the laboratory must not report a quantitative result for a target analyte that was not included in the calibration standard(s).**
- **Internal Standards:** The calibration standards must also contain the internal standards chosen for the analysis. Calibration standards for soils must also contain the preservative Na_2SO_4. See Method 5035 / NV05-108 for how to accomplish the preservation.
- **Surrogates**: Historically the surrogate compounds have been included in the multi-point initial calibration at variable concentrations in order to evaluate the linear response as with any target analyte. With improvements in instrumentation and more reliance on the autosampler, an option is available allowing the autosampler to spike the initial calibration standards with surrogates in the same manner as the samples are spiked. With this option, the surrogate standards in the initial calibration can be averaged to develop a response factor and an effective one-point calibration with the sole purpose to measure the surrogate recovery using the same concentration for each sample analysis. For this calibration option, the surrogate linear response is less important, since multiple concentrations of surrogates are not being measured. Instead, the surrogate concentration remains constant throughout, and the recovery of this known concentration can easily be attained without demonstrating if the response is linear.
- **Technique:** To prepare a calibration standard, add an appropriate volume of a secondary dilution standard solution to an aliquot of organic-free reagent water in a Class A, volumetric flask. Use a microsyringe and rapidly inject the alcoholic standard into the expanded area of the filled volumetric flask. Remove the needle as quickly as possible after injection and stopper. Mix by inverting the flask three times. Discard the contents contained in the neck of the flask. Aqueous standards are not stable and are prepared daily. Transfer each standard to separate VOA vials.
- **Water or Soil Samples:** A different calibration curve is necessary for Methods 5030 / NV05-107 and 5035 / NV05-108. Calibration **must** be performed using the same sample introduction technique that is used for samples. For Method 5030, the purging efficiency varies with purge volume; therefore, develop the standard curve with whichever volume of sample that is to be analyzed.

3 **Calibration sequence**:

1	BFB Tuning Criteria

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		2	ICAL
		3	ICV
		4	ICB
4	**Tabulate the area response of the characteristic ions** (see Attachment 1) against the concentration for each target analyte and each internal standard. Calculate response factors (RF) for each target analyte relative to one of the internal standards.		
5	Evaluate the **linearity of target analytes** • If the RSD of any target analyte is **15%** or less, then the response factor is assumed to be constant over the calibration range, and the average response factor is used for quantitation. • If the RSD of any target analyte is greater than 15%, linear regression is used. The correlation coefficient (r^2) must be at least 0.99 ($r \geq 0.995$).		
6	Evaluate the SPCC and CCC compounds for the initial calibration criteria.		
7	Evaluate the retention times and minimum response factors.		
8	Evaluate the success of the initial calibration by immediately running an Initial Calibration Verification (ICV).		
9	Evaluate the Initial Calibration Blank to be sure it is free of contaminants.		

10.3 Daily GC/MS Calibration Verification

1	Evaluate the BFB tuning criteria.
2	Evaluate the CCV and CCB.
3	Evaluate the SPCC and CCC compounds for the continuing calibration criteria.

10.4 Example Analysis Queue / Sequence (based on 12 hours)

1	Tune
2	CCV, for daily and ongoing calibration check
3	LCS
4	Blank
5	Samples
6	Matrix Spike
7	Matrix Spike Duplicate
When 12 hours have passed, run a 2nd tune and CCV before running more samples, no more than 20 samples in a 12-hour batch.	

Note: For samples from the states of Oklahoma and Wyoming, QC (blank, MS/MSD, and LCS) **must** be done in batches of no more than 10 samples.

- **Dilutions**: If the initial analysis of the sample or a dilution of the sample has a concentration of any analyte that exceeds the upper calibration standard, the sample must be reanalyzed at a higher dilution. **Secondary ion quantitation is allowed only when there are sample interferences with the primary ion**.
  - When ions from a compound in the sample saturate the detector, this analysis must be followed by the analysis of an organic-free, reagent water blank or the repeating of suspected samples. If the blank analysis is not free of interferences, then the system must be decontaminated. Sample analysis may not resume until the blank analysis is demonstrated to be free of interferences. Repeat all affected samples.
  - Prepare dilutions such that the response of the major constituents (previously saturated

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peaks) is in the upper half of the linear range of the curve.

- The following procedure is used to dilute aqueous samples for analysis of volatiles. All steps must be performed without delays.

1	Dilutions are made in Class A, volumetric flasks (50 to 100 mL). Select the volumetric flask that allows for the necessary dilution. Intermediate dilution steps may be necessary for extremely large dilutions.
2	Calculate the approximate volume of organic-free reagent water to be added to the volumetric flask, and add slightly less than this quantity of organic-free reagent water to the flask.
3	Inject the appropriate volume of the original sample from the syringe into the flask. Aliquots of less than 1 mL are not recommended. Dilute the sample to the mark with organic-free reagent water. Cap the flask, invert, three times. Repeat above procedure for additional dilutions.
4	Fill a VOA vial with the diluted sample and cap.

- For high concentration samples, see SOP 5035 / NV05-108.

10.5 Qualitative analysis

The qualitative identification of each compound determined by this method is based on relative retention time, and on comparison of the sample mass spectrum, after background correction, with characteristic ions in a reference mass spectrum. The reference mass spectrum must be kept up to date and obtained through analysis of known standards on the instrument using the conditions of this method. The characteristic ions from the reference mass spectrum are defined to be the three ions of greatest relative intensity, or any ions over 30% relative intensity if less than three such ions occur in the reference spectrum. See Attachment 1 for primary and secondary ions for each compound. Compounds are identified as present when the following criteria are met:

- The intensities of the characteristic ions of a compound maximize in the same scan or within one scan of each other. Selection of a peak by a data system target compound search routine where the search is based on the presence of a target chromatographic peak containing ions specific for the target compound at a compound-specific retention time is accepted as meeting this criterion.
- The relative retention time (RRT) of the sample component is within ± 0.06 RRT units of the RRT of the standard component.
- The relative intensities of the characteristic ions agree with 30% of the relative intensities of these ions in the reference spectrum. (Example: For an ion with an abundance of 50% in the reference spectrum, the corresponding abundance in a sample spectrum can range between 20% and 80%). When two or more analytes that co-elute share secondary ions, and all the characteristic secondary ions for the target analyte are present but outside the ± 30 % relative intensity, report the compound as positive if there is no interference with the primary quantitation ion. If co-eluting peaks share the primary ion, the analyte may only be reported as a co-eluting pair.
- Identification is hampered when sample components are not resolved chromatographically and produce mass spectra containing ions contributed by more than one analyte. When gas chromatographic peaks obviously represent more than one sample component (i. e., a broadened peak with shoulder(s) or a valley between two or more maxima), appropriate selection of analyte spectra and background spectra is important.
- Examination of extracted ion current profiles of appropriate ions can aid in the selection of spectra, and in qualitative identification of compounds. When analytes co-elute (i. e., only one chromatographic peak is apparent), the identification criteria may be met, but each analyte spectrum might contain extraneous ions contributed by the co-eluting compound. If all of the

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ions associate with the reference spectrum for the target analyte are present and within the ± 30% criteria, a positive result must be assumed even in the presence of extraneous ion fragments without presumptive evidence for a negative identification. (All ions associated with the target analyte are also present in the interfering peak.) The analyst must carefully weigh the background spectrum and the spectrum of any co-eluting analytes whenever assessing a potential hit. Analyst experience in interpreting mass spectral data and the above specified guidelines are used together to interpret difficult matrices. Add appropriate qualifiers in Element (ID2).

- Structural isomers that produce very similar mass spectra are identified as individual isomers if they have sufficiently different GC retention times. Sufficient GC resolution is achieved if the height of the valley between two isomer peaks is less than 25% of the sum of the two peak heights. Otherwise, structural isomers are identified as isomeric pairs.
- For samples containing components not associated with the calibration standards, a library search may be made for the purpose of tentative identification. The necessity to perform this type of identification is determined by the purpose of the analyses being conducted. Data system library search routines are not to use normalization routines that would misrepresent the library or unknown spectra when compared to each other.
- For example, the RCRA permit or waste delisting requirements may require the reporting of non-target analytes. Only after visual comparison of sample spectra with the nearest library searches may the analyst assign a tentative identification. Use the following guidelines for making tentative identifications:
  - Relative intensities of major ions in the reference spectrum (ions greater than 10% of the most abundant ion) must be present in the sample spectrum.
  - The relative intensities of the major ions must agree within ± 20%. (Example: For an ion with an abundance of 50% in the standard spectrum, the corresponding sample ion abundance must be between 30 and 70%).
  - Molecular ions present in the reference spectrum must be present in the sample spectrum.
  - Review ions present in the sample spectrum but not in the reference spectrum for possible background contamination or presence of co-eluting compounds.
  - Review ions present in the reference spectrum but not in the sample spectrum for possible subtraction from the sample spectrum because of background contamination or co-eluting peaks. Data system library reduction programs can sometimes create these discrepancies.

10.6 Quantitative analysis

- Once a compound has been identified, the quantitation of that compound is based on the integrated abundance from the EICP of the **primary** characteristic ion. The internal standard used is the one nearest the retention time of that of a given analyte. See Attachment 1.
- If the RSD of a compound's response factors is 15% or less, then the concentration is determined using the average response factor (*RF*) from initial calibration data.
- Where applicable, the concentration of any non-target analyte identified in the sample may be estimated. The same formulae are used with the following modifications: The areas A_x and A_{is} are from the total ion chromatograms, and the RF for the compound must be assumed to be 1.
- The resulting concentration is reported indicating:
  - that the value is an estimate, and
  - in Level 4 data packages, which internal standard was used to determine the concentration. Use the nearest internal standard free of interferences.

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11.0 Calculations / Data Reduction

11.1 Accuracy

$$\% \text{ Recovery} = \frac{\text{Measured concentration}}{\text{Known concentration}} \times 100$$

11.2 Precision (RPD)

$$\text{RPD} = \frac{\text{Absolute value (orig. sample value - dup. sample value)}}{\text{(Orig. sample value + dup. sample value)/2}} \times 100$$

11.3 Response Factor

$$RF = \frac{A_s x C_{is}}{A_{is} x C_s}$$

A_s = Peak area of the analyte or surrogate.
A_{is} = Peak area of the internal standard.
C_s = Concentration of the analyte or surrogate.
C_{is} = Concentration of the internal standard

11.4 Standard Deviation, Relative Standard Deviation

$$SD = \sqrt{\frac{\sum_{i=1}^{n} (RF_i - RF_{mean})^2}{n-1}} \qquad RSD = \frac{SD \times 100}{RF_{mean}}$$

RF_i = RF for each of the calibration standards
RF_{mean} = mean RF for each compound from the initial calibration
n = Number of calibration standards, e. g., 5

11.5 % Difference, % Drift

$$\% \text{ Difference} = \frac{(RF_v) - (\text{Avg. RF})}{(\text{Avg. RF})} \times 100$$

RF_v = RF from verification standard
Avg. RF = Average RF from Initial Calibration.

$$\% \text{ Drift} = \frac{\text{Result - True Value}}{\text{True Value}} \times 100$$

11.6 Linear Calibration Using a Least Squares Regression: A linear calibration model based on a least squares regression may only be employed if RSD does not meet the acceptance criteria.

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For calibration, x is the mass of the analyte in the sample aliquot introduced into the instrument and y is the area (or height) or the response, as in:

$x = C_s$ and $y = A_s$

A linear least squares regression attempts to construct a linear equation of the form:

$y = ax + b$

by minimizing the differences between the observed results (y_i, the instrument response) and the predicted results (y_i', the response calculated from the constructed equation). The regression equation is:

$y_i' = ax_i + b$

a = regression coefficient or the slope of the line.
b = the y-intercept.
y_i' = predicted (or calculated) response for the i^{th} calibration standard.
x_i = mass of analyte in the i^{th} calibration standard aliquot introduced into the instrument.

The sum of the squares of the differences is minimized to obtain a and b:

$$\sum_{i=1}^{n}(y_i - y_i')^2$$

n = total number of calibration points. The regression calculations attempt to minimize this sum of the squares, hence the name "least squares regression."

Weighting the sum of the square of the differences may significantly improve the ability of the least squares regression to fit the linear model to the data. The general form of the sum of the squares of the differences containing the weighting factor is:

$$\sum_{i=1}^{n} w_i(y_i - y_i')^2$$

w_i = weighting factor for the i^{th} calibration standard (w=1 for unweighted least squares regression).
y_i – observed instrument response (area) for the i^{th} calibration standard.
y_i' = predicted (or calculated) response for the i^{th} calibration standard.
n = total number of calibration standards.

The mathematics used in least squares regression has a tendency to favor numbers of larger value over numbers of smaller value. Thus the regression curves that are generated tend to fit points that are at the upper calibration levels better than those points at the lower calibration levels. To compensate for this, a weighting factor which reduces this tendency can be used. Examples of allowed weighting factors which place more emphasis on numbers of smaller value are:

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$w_i - 1/y_i$ or $w_i = 1/y_i^2$

Do not include the origin (0, 0) as an extra calibration point. The use of a linear regression may NOT be used as a rationale for reporting results below the calibration range demonstrated by the analysis of the standards. If it is necessary to report results at lower concentrations, then the analyst must run a calibration that reaches those lower concentrations.

The regression calculation generates a correlation coefficient (r) that is a measure of the "goodness of fit" of the regression line to the data. A value of 1.00 indicates a perfect fit. In order to be used for quantitative purposes, r must be greater than or equal to 0.995 or $r^2 \geq 0.99$.

11.7 Coefficient of Determination **Correlation Coefficient**

$$r^2 = \frac{\left(\sum xy\right)^2}{\sum x^2 \sum y^2} \qquad r = \frac{\left(\sum xy\right)}{\sqrt{\sum x^2 \sum y^2}}$$

y = Response ratio
x = Concentration

11.8 Concentration Calculation

Concentration = (µg/L from instrument) (dilution factor)

12.0 Method Performance

12.1 Method Detection Limit Study (MDL): The method detection limit (MDL) is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. The MDL is determined according to the laboratory's MDL procedure in SOP Determination of Method Detection Limits / NV08-202. MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix, and may not be achievable in all environmental matrices. The laboratory maintains MDL studies for analyses performed; these are verified at least annually unless method requirements require a greater frequency.

12.2 Demonstration of Capability: The laboratory demonstrates initial proficiency by generating data of acceptable accuracy and precision for target analyses in a clean matrix. The laboratory also repeats the operation whenever new staff is trained or significant changes in instrumentation are made and on an annual basis thereafter. See the training section of TestAmerica-Nashville's QA Manual and SOP Training / NV08-199 for information on how to accomplish this demonstration.

12.3 Training Requirements: Demonstration of Capability is performed initially when learning the method and annually thereafter. Four Laboratory Control Samples resulting in an average % recovery within the control limits and a precision less than the quality control maximum are required.

12.4 Proficiency Testing Studies: The laboratory participates in formal proficiency testing (PT) studies, where solutions of unknown concentrations are analyzed and the performance of all participants is compared. See the QA department for the results of recent PT studies.

13.0 Pollution Control

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It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i. e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention."

14.0 Waste Management

14.1 Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes must be stored, managed, and disposed of in accordance with all federal and state laws and regulations. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to the QA Manual and SOP Waste Disposal / NV10-83.

14.2 Wastestreams Produced by the Method:

- Aqueous waste generated from analysis may have a pH of less than 2.0. Transfer to waste disposal for neutralization and then dump into the sanitary sewer.
- Solid waste generated from analysis is placed in the trash.

15.0 References / Cross References

15.1 EPA Method 8260B, SW-846 Update III, Revision 2, December 1999.

15.2 Method 8000B, SW-846, Revision 2, December 1996, Method 8000C, Revision 3, March 2003.

15.3 Method TPH-GRO by Method 8260B, MRBCA (Missouri) Guidance Document, Final Draft, February 24, 2004.

15.4 California GRO, CA LUFT 8015.

15.5 TestAmerica Nashville's Quality Assurance Manual.

15.6 TestAmerica Nashville's Control Limits Manual.

15.7 Corporate Environmental Health and Safety Manual (CW-E-M-001).

15.8 SOPs: Manual Integration / CA-Q-S-002, Selection of Calibration Points / CA-T-P-002, Waste Disposal / NV10-83, Training Procedures for Environmental Technical Staff / NV08-199, Determination of Method Detection Limits / NV08-202, Reagent and Standard Purchase / NV08-214, Subsampling & Compositing / NV08-229, 5030 / NV05-107, 5035 / NV05-108.

15.9 Controlled Document: QAF-45, TestAmerica Nashville – Acronyms, Keywords, and Definitions.

16.0 Method Modifications

State	Modification
Ohio specific criteria	Only those compounds in EPA Method 8260B may be reported (superscript 1 in the table in Section 1.1). Some compounds in this SOP are not part of the original 8260B method. The method blank must be less than the RL for Ohio samples.
Missouri GRO	Prepare 1:1 mixture of unleaded gasoline and #2 diesel fuel in Methanol as GRO is defined by setting retention time window from 0.1 minutes before C_6 to 0.1 minutes after C_{10}. Verify RT window with the standard daily (every 24 hours).
California GRO	California LUFT GRO uses gasoline and the retention time window of C_4 (t-butanol) to C_{12}.
Michigan GRO	See the Michigan GRO requirements below.

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TPH-GRO by Method 8260B

Sample Introduction

- Samples are purged onto the GC/MS system using all protocols specified in SW-846 Method 5030 and 5035.
- Surrogates and internal standards specified by Method 8260B are added to water and soil samples prior to purging.

Sample Analysis

- The BC/MS system is tuned to BFB tune criteria listed in Method 8260B at the frequency specified in Method 8260B.
- A 5-point standard curve is used to quantitate TPH-GRO by the external standard technique.
- For **Missouri** GRO, the stock standard solution is a mixture of unleaded gasoline and Number 2 diesel fuel.
- For **California** GRO, the stock standard is unleaded gasoline.
- The lowest calibration standard should be at or below the reporting limit of the method.
- For **Missouri,** retention time windows are defined for TPH-GRO by analyzing a standard containing C_6 and C_{10}. The retention time window is defined as $>C_6$ to C_{10}. The standard containing C_6 and C_{10} must be analyzed every day samples are analyzed in order to verify that the retention time windows are constant.
- For **California**, the retention time window are defined as $>C_4$ to C_{12}.
- For **Michigan**,
  - Use unleaded gasoline for calibration.
  - The retention time window is defined as C_6 (hexane) to C_{10} (n-decane).
  - The holding time for water and soil is 14 days.
  - For soil preparation, shake the methanol and sample for 2 minutes, then sonicate in a water bath for 20 minutes.
  - For oil samples, add 2 g sample to 40 mL methanol; wait 24 hours before analysis.
  - Use the external calibration technique, summing the range.
  - Use only linear regression; $r \geq 0.99$, $r^2 \geq 0.981$.
  - ICV and CCV must be ± 20% true.
- Because the retention time window is several minutes wide for TPH-GRO, the GC/MS data system may not accurately or appropriately establish the proper baseline for calibration or quantitation. The analyst **must** visually examine the computer-generated baseline for every analytical run and manually adjust the baseline when needed. A properly drawn baseline must extend over the entire retention time window and include the area under the entire TPH-GRO series of peaks. It is not appropriate to draw the baseline "peak to peak."
- The total ion chromatogram (TIC) **must** be used to calculate the area under the peak for TPH-GRO calibration and quantitation determinations over the entire retention time window.
- Area counts for the internal standards and surrogates added during sample preparation **must** be subtracted from the total area count for TPH-GRO. This is accomplished by subtracting the area count of the method blank from all subsequent calibration and analytical runs.
- The %RSD for the calibration curve for TPH-GRO must be less than or equal to 20%, so that linearity through the origin can be assumed and an average calibration factor used for calculations.

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- A continuing calibration check standard (CCC) must be analyzed every 12 hours of sample analysis. The standard concentration should be at the mid-point of the calibration curve. If the %RSD of the second CCC exceeds 20%, a new curve must be generated.
- A method blank must be analyzed once per day to insure the analytical system is free of background contamination.

17.0 Attachments

17.1 Attachment 1, Characteristic Masses (m/z) for Purgeable Organic Compounds.

17.2 Attachment 2, Volatile Internal Standards with Corresponding Analytes Assigned for Quantitation.

17.3 Attachment 3, South Carolina LCS acceptance criteria.

Attachment 1, Characteristic Masses (m/z) for Purgeable Organic Compounds

Compound	Primary Characteristic Ion	Secondary Characteristic Ion(s)
Acetone	58	43
Acetonitrile	41	40, 39
Acrolein	56	55
Acrylonitrile	53	52, 51
Allyl chloride	76	78
Benzene	78	-
Benzyl chloride	91	126, 65, 128
Bromobenzene	77	156, 158
Bromochloromethane	130	49, 128
Bromodichloromethane	83	85, 127
Bromoethane	108	110
Bromoform	173	175, 254
Bromomethane	96	94
2-Butanone	72	43
Butyl acetate	43	56, 73, 61
n-Butyl alcohol	56	41, 43
t-butyl alcohol	59	41, 43
n-Butylbenzene	91	92, 134
sec-Butylbenzene	105	134
tert-Butylbenzene	119	91, 134
Carbon disulfide	76	78
Carbon tetrachloride	117	119
Chlorobenzene	112	77, 114
Chloroethane	64	66 (51*)
2-Chloroethanol	49	44, 43, 51, 80
2-Chloroethyl vinyl ether	63	65, 106
Chloroform	83	85
1-chlorohexane	91	55, 41
Chloromethane	50	52 (51*)
Chloroprene	53	88, 90, 51
2-Chlorotoluene	91	126
4-Chlorotoluene	91	126
Cyclohexanone	55	42, 98
Cyclohexane	56	84, 41, 69
Dibromochloromethane	127	129
1,2-Dibromo-3-chloropropane	157	155, 75

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Compound	Primary Characteristic Ion	Secondary Characteristic Ion(s)
1,2-Dibromoethane	107	109, 188
Dibromomethane	93	95, 174
2,6-Dimethyl-4-heptone (DIBK)	57	85, 142
1,2-Dichlorobenzene	146	111,148
1,3-Dichlorobenzene	146	111, 148
1,4-Dichlorobenzene	146	111, 148
cis-1,4-Dichloro-2-butene	53	88, 89
trans-1,4-Dichloro-2-butene	53	88, 89
Dichlorodifluoromethane	85	87
1,1-Dichloroethane	63	65, 83
1,2-Dichloroethane	62	98
1,1-Dichloroethene	96	61, 63
cis-1,2-Dichloroethene	61	96, 98
trans-1,2-Dichloroethene	61	96, 98
Dichlorofluoromethane	67	69
1,2-Dichloropropane	63	112
1,3-Dichloropropane	76	78
2,2-Dichloropropane	77	97
1,1-Dichloropropene	75	110, 77
cis-1,3-Dichloropropene	75	77, 39
trans-1,3-Dichloropropene	75	77, 39
2,3-Dichloro-1-propene	75	39, 110
Diethyl ether	59	45, 74
3,3-Dimethyl-1-butanol	57	69, 41
1,4-Dioxane	88	58, 43, 57
Ethanol	45	46
Ethyl acetate	43	45, 61, 88
Ethylbenzene	91	106
Ethyl methacrylate	69	41, 99, 86
ETBE	59	87, 41
ETBA	59	45, 87, 43
Hexachlorobutadiene	225	223, 227
Hexane	57	41, 43, 56
2-hexanone	58	43, 57, 100
Iodomethane	142	127, 141
Isobutyl alcohol	43	41, 42, 74
Isobutylisobutyrate	71	43, 56
Isopropyl alcohol	45	59
Isopropylbenzene	105	120
Isopropylether (IPE)	45	87, 59
p-Isopropyltoluene	119	134, 91
Methacrylonitrile	41	67, 39, 52
Methyl acetate	43	74, 59
Methyl cyclohexane	83	55, 98, 41
Methyl-t-butyl ether	73	57, 43
Methylene chloride	84	86, 49
Methyl methacrylate	41	69, 100, 39
1-Methylnaphthalene	142	141, 115
2-Methylnaphthalene	142	141, 115
4-Methyl-2-pentanone	58	43, 100, 85

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Compound	Primary Characteristic Ion	Secondary Characteristic Ion(s)
Naphthalene	128	-
2-Nitropropane	43	41, 39
Nonane	43	57, 85, 128
Octane	43	57, 85, 114
Pentane	43	41, 57, 72
Propionitrile (ethyl cyanide)	54	52, 55
n-Propylbenzene	91	120
Styrene	104	78
TBF	59	57, 41
TAA	59	55, 73, 43
TAME	73	55, 87, 43
1,1,1,2-Tetrachloroethane	131	133, 119
1,1,2,2-Tetrachloroethane	83	131, 85
Tetrachloroethene	166	129, 131, 164
Tetrahydrofuran	42	41, 71, 72
Toluene	91	92
1,2,3-Trichlorobenzene	180	182, 145
1,2,4-Trichlorobenzene	180	182, 145
1,3,5-Trichlorobenzene	180	145, 182
1,1,1-Trichloroethane	97	99, 61
1,1,2-Trichloroethane	97	83, 85
Trichloroethene	130	97, 95, 132
Trichlorofluoromethane	101	103, 105
1,2,3-Trichloropropane	110	75, 77
Trichlorotrifluoroethane	101	151, 103, 153
1,2,4-Trimethylbenzene	105	120
1,3,5-Trimethylbenzene	105	120
Vinyl acetate	43	86
Vinyl chloride	62	64
o-Xylene	91	106
m-Xylene	91	106
p-Xylene	91	106
Internal Standards/Surrogates:		
Fluorobenzene	96	70
Chlorobenzene-d_5	117	82
1,4-Dichlorobenzene-d_4	152	115, 78
4-Bromofluorobenzene	95	174, 176
Dibromofluoromethane	111	113
1,2-Dichloroethane-d_4	65	67, 51
Toluene-d_8	98	100

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Attachment 2, Volatile Internal Standards with Corresponding Analytes Assigned for Quantitation

Fluorobenzene	Chlorobenzene-d_5	1,4-Dichlorobenzene-d_4
Dichlorodifluoromethane	2-Chloroethylvinylether	Bromobenzene
Chloromethane	cis-1,3-Dichloropropene	1,1,2,2-Tetrachloroethane
Vinyl chloride	ETBA	1,2,3-Trichloropropane
Bromomethane	4-Methyl-2-pentanone	trans-1,4-Dichloro-2-butene
Chloroethane	Toluene	Bromofluorobenzene (s)

Fluorobenzene	**Chlorobenzene-d_5**	**1,4-Dichlorobenzene-d_4**
Dichlorofluoromethane	trans-1,3-Dichloropropene	Propylbenzene
Trichlorofluoromethane	Ethyl methacrylate	2-Chlorotoluene
Ethanol	1,1,2-Trichloroethane	4-Chlorotoluene
Diethyl ether	Toluene-d8 (s)	1,3,5-Trimethylbenzene
Acrolein	Tetrachoroethene	t-Buylbenzene
Trichlorotrifluoroethane	1,3-Dichloropropane	1,2,4-Trimethylbenzene
1,1-Dichloroethene	2-Hexanone	sec-Butylbenzene
Acetone	Dibromochloromethane	p-Isopropyltoluene
Iodomethane	1,2-Dibromoethane	1,3-Dichlorobenzene
Isopropyl alcohol	Chlorobenzene	1,4-Dichlorobenzene
Carbon disulfide	1,1,1,2-Tetrachloroethane	1,2-Dichlorobenzene
Methyl acetate	Ethylbenzene	Butylbenzene
Methylene chloride	m,p-Xylene	1,2-Dibromo-3-chloropropane
t-Butyl alcohol	o-Xylene	1,2,4-Trichlorobenzene
Acrylonitrile	Styrene	Hexachlorobutadiene
trans-1,2-Dichloroethene	Bromoform	Naphthalene
MTBE	Isopropylbenzene	1,2,3-Trichlorobenzene
1,1-Dichloroethane	DIBK	2-Methylnaphthalene
Vinyl acetate	Benzyl chloride	1,3,5-Trichlorobenzene
Diisopropyl ether	3,3-Dimethyl-1-butanol	1-Methylnaphthalene
ETBE	Nonane	
2,2-Dichloropropane	Octane	
cis-1,2-Dichloroethene		
2-Butanone		
Bromochloromethane		
Chloroform		
t-Butyl formate		
1,1,1-Trichloroethane		
Cyclohexane		
1,2-Dichloroethane-d_4 (s)		
Dibromofluoromethane (s)		
Carbon tetrachloride		
1,1-Dichloropropene		
Benzene		
1,2-Dichloroethane		
t-Amyl alcohol		
TAME		
Trichloroethene		
n-Butanol		
Methyl cyclohexane		
1,2-Dichloropropane		
2,3-Dichloro-1-Propene		
Dibromomethane		
Bromodichloromethane		
1,4-Dioxane		
Acetonitrile		
Bromoethane		
Ally chloride		
Propionitrile		
Chloroprene		
Hexane		

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Fluorobenzene	Chlorobenzene-d_5	1,4-Dichlorobenzene-d_4
Methacrylonitrile		
Tetrahydrofuran		
Isobutanol		
Heptane		
2-Nitropropane		
Methyl methacrylate		
Butyl acetate		
1-Chlorohexane		
Isobtylisobutyrate		
cis-1,4-Dichloro-2-butene		
Cyclohexanone		
Pentane		

Attachment 3, South Carolina LCS Acceptance Criteria and Special State PQL Requirements

- All routinely reported analytes require 70-130% LCS recovery. The following exceptions of poor-performing analytes require 60-140% LCS recovery:

Acetonitrile	Dichlorodifluoromethane
Acrolein	2,2-Dichloropropane
Acrylonitrile	3,3-Dimethyl-1-butanol
t-Amyl alcohol	1,4-Dioxane
Bromomethane	Ethanol
t-Butyl alcohol	2-Hexanone
t-Butyl formate	Isopropyl alcohol
Chloroethane	4-Methyl-2-pentanone
Chloromethane	Vinyl chloride
1,2-Dibromo-3-chloropropane	

- Instrumentation used for South Carolina samples must be able to achieve and report the following South Carolina PQLs when those compounds are requested:
  - Acrolein 5 ug/L
  - Acrylonitrile 5 ug/L
  - 2-Chloroethyl vinyl ether 5 ug/L
  - Methylene Chloride 2 ug/L

18.0 Revision History

- Revision 12, 10 October 2008
  - Integration for TestAmerica and STL operations.
  - Insert corrective action procedures
- Revision 13, 25 September 2009
  - Move QC summary table and QC sample preparation instructions into Section 9.
  - Addition of new analytes: 3,3-Dimethyl-1-butanol (SC) and 1,3,5-Trichlorobenzene (NH).
  - Addition of Attachment 3 for South Carolina.
- Revision 14, 6 November 2009
  - Corporate review.
  - Addition of single-point surrogate calibration.
  - Incorporate Michigan GRO requirements.
- Revision 15, 30 October 2010

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- Addition of Amendments a (SC PQLs, Attachment 3), b (characteristic ions for 1,2,3-Trichloropropane), and c (WI soil extraction procedure).
- Addition of new analytes: 1-Methylnaphthalene (New Mexico), Pentane, Octane, Nonane (Paraffin group).
- Addition of QAF-45 and Section 14.2.

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Title: POLYCHLORINATED BIPHENYLS (PCBs) BY GAS CHROMATOGRAPHY EPA METHOD 8082 / 8082A

Approvals (Signature/Date):

Cory Spry — 10/26/10
Department Supervisor — Date

Michael H. Dunn — 9-20-10
Technical Director — Date

Jared Knierim — 10-26-10
Health & Safety Manager / Coordinator — Date

Eric S. Smith — 10/29/10
Quality Assurance Manager — Date

Johnny A. Mitchell — 10.26.10
Laboratory Director — Date

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1.0 Scope and Application

1.1 Analyte, Matrices: This method is used to determine the concentrations of polychlorinated biphenyls (PCBs) as Aroclors in extracts from solid, oil, and aqueous matrices. Open-tubular, capillary columns are employed with electron capture detectors (ECD). The target compounds listed below may be determined by the dual-column analysis system.

Compound	CAS No.	Water RL (µg/L)	Soil RL (µg/kg)	Oil RL (µg/kg)
Aroclor 1016	12674-11-2	0.5	16.67	16.67
Aroclor 1221	11104-28-2	0.5	16.67	16.67
Aroclor 1232	11141-16-5	0.5	16.67	16.67
Aroclor 1242	53469-21-9	0.5	16.67	16.67
Aroclor 1248	12672-29-6	0.5	16.67	16.67
Aroclor 1254	11097-69-1	0.5	16.67	16.67
Aroclor 1260	11096-82-5	0.5	16.67	16.67
Aroclor 1262	37324-23-5	0.5	16.67	16.67
Aroclor 1268	11100-14-4	0.5	16.67	16.67

1.2 Reporting Limits for water and soil are shown in the table above.

1.3 Aroclors are multi-component mixtures. When samples contain more than one Aroclor, a higher level of analyst expertise is required to attain acceptable levels of qualitative and quantitative analysis. The same is true of Aroclors that have been subjected to environmental degradation ("weathering") or degradation by treatment technologies. Such **weathered multi-component mixtures may have significant differences in peak patterns than those of Aroclor standards**.

1.4 This method describes a dual-column procedure with a hardware configuration of two analytical columns joined to a single injection port.

1.5 This method is restricted to use by or under the supervision of analysts experienced in the use of a gas chromatograph and in the interpretation of gas chromatograms. Each analyst demonstrates the ability to generate acceptable results with this method.

1.6 If for any reason a part of this method cannot be followed, seek the guidance of the Department Supervisor or the Laboratory Technical Director. All abnormalities must be noted on the data or the benchsheet and in the Laboratory Information Management System (LIMS).

2.0 Summary of Method

2.1 A measured volume or weight of sample (approximately 500 mL for liquids, 30 g for solids, 1 g for oil) is extracted using the appropriate matrix-specific sample extraction technique.

2.2 Aqueous samples are extracted at neutral pH with methylene chloride using Method 3510 / NV03-24 / (separatory funnel).

2.3 Solid samples are extracted with methylene chloride using Method 3550 / NV03-23 (ultrasonic) or 3541 / NV03-231 (automated Soxhlet). Oils are diluted using Method 3580 / NV03-106.

2.4 Extracts for PCB analyses are subjected to a sulfuric acid cleanup Method 3665 / NV03-117 designed specifically for these analytes.

2.5 After cleanup, the extract is analyzed by injecting a nominal 1 uL aliquot into a gas chromatograph with narrow-bore fused silica capillary columns and electron capture detectors (GC/ECD).

3.0 Definitions

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See TestAmerica Nashville's Quality Assurance Manual Appendix 5 for laboratory definitions. Also, refer to Controlled Document QAF-45, TestAmerica Nashville Acronyms, Keywords, and Definitions.

4.0 Interferences

4.1 Contamination by carryover can occur whenever high-concentration and low-concentration samples are analyzed in sequence. To reduce the potential for carryover, the sample syringe is thoroughly rinsed between samples with hexane.

4.2 Interferences co-extracted from the samples vary considerably from matrix to matrix. While general cleanup techniques are referenced or provided as part of this method, unique samples may require additional cleanup approaches to achieve desired degrees of discrimination and quantitation. Sources of interference in this method can be grouped into three broad categories:

- Contaminated solvents, reagents, or sample processing hardware.
- Contaminated GC carrier gas, parts, column surfaces, or detector surfaces.
- Compounds extracted from the sample matrix to which the detector responds.

4.3 Interferences by phthalate esters introduced during sample preparation can pose a major problem in PCB determinations.

- Common flexible plastics contain varying amounts of phthalate esters which are easily extracted or leached from such materials during laboratory operations. Interferences from phthalate esters can best be minimized by avoiding contact with any plastic materials and checking all solvents and reagents for phthalate contamination.
- Exhaustive cleanup of solvents, reagents and glassware may be required to eliminate background phthalate ester contamination.
- These materials can be removed through the use of Method 3665 / NV03-117 (sulfuric acid cleanup).

4.4 Cross-contamination of clean glassware routinely occurs when plastics are handled during extraction steps, especially when solvent-wetted surfaces are handled. Glassware is scrupulously cleaned. Clean all glassware as soon as possible after use by detergent washing with hot water, and rinses with tap water. Drain the glassware, and rinse with acetone. Store dry glassware in a clean environment.

4.5 Elemental sulfur (S_8) is readily extracted from soil samples and may cause chromatographic interferences in the determination of PCBs. Sulfur can be removed through the use of Method 3660 /NV03-99.

5.0 Safety

Employees must abide by the policies and procedures in the Corporate Safety Manual, Radiation Safety Manual and this document. This procedure may involve hazardous material, operations and equipment. This method does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.1 Specific Safety Concerns or Requirements:

- The gas chromatograph contains zones that have elevated temperatures. The analyst needs to be aware of the locations of those zones, and must cool them to room temperature prior to working on them.

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- There are areas of high voltage in the gas chromatograph. Depending on the type of work involved, either turn the power to the instrument off, or disconnect it from its source of power.

5.2 Primary Materials Used: The following is a list of the materials used in this method, which have a serious or significant hazard rating. **Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material	Hazards	Exposure Limit (1)	Signs and symptoms of exposure
Acetone	Flammable	1000 ppm-TWA	Inhalation of vapors irritates the respiratory tract. May cause coughing, dizziness, dullness, and headache.
Methylene Chloride	Carcinogen Irritant	25 ppm-TWA 125 ppm-STEL	Causes irritation to respiratory tract. Has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting and headache. Causes irritation, redness and pain to the skin and eyes. Prolonged contact can cause burns. Liquid degreases the skin. May be absorbed through skin.
Hexane	Flammable Irritant	500 ppm-TWA	Inhalation of vapors irritates the respiratory tract. Overexposure may cause lightheadedness, nausea, headache, and blurred vision. Vapors may cause irritation to the skin and eyes.
Sulfuric Acid	Corrosive Oxidizer Dehydrator Poison Carcinogen	1 mg/m^3-TWA	Inhalation produces damaging effects on the mucous membranes and upper respiratory tract. Symptoms may include irritation of the nose and throat, and labored breathing. Symptoms of redness, pain, and severe burn can occur. Contact can cause blurred vision, redness, pain and severe tissue burns. Can cause blindness.
1 – Exposure limit refers to the OSHA regulatory exposure limit.			

6.0 Equipment and Supplies

6.1 Instrumentation

- Gas chromatograph (Perkin-Elmer, Shimadzu, Hewlett-Packard) - An analytical system complete with gas chromatograph suitable for on-column injection and all required accessories including syringes, analytical columns, gases, electron capture detectors (ECD), and data system (EZChrom Data Acquisition System).
- GC columns: This method describes procedures for dual-column analyses. The dual-column approach employs two (0.32 mm ID) columns. Other capillary columns, provided that they document method performance (e.g., chromatographic resolution, analyte breakdown, and MDLs) that equals or exceeds the performance specified in this method, may be used. Record any changes in the maintenance logbook.
  - Column pair 1: 30 m x 0.32 mm ID fused silica capillary column chemically bonded with 5% diphenyl / 95% dimethyl polysiloxane (Restek Rtx-5, Rtx-CLPesticides, or equivalent), 0.5 µm film thickness. 30 m x 0.32 mm ID fused silica capillary column chemically bonded with 50% methyl / 50 % phenyl polysiloxane (Rtx-50, Rtx-CLPesticides 2, or equivalent), 0.5 µm film thickness.
  - Column pair 1 is mounted in a press-fit Y-shaped fused-silica connector (Restek, Catalog No. 20405), or equivalent.
  - Guard column : 5 m x 0.53 mm, deactivated
- **GC Conditions**: Use the conditions in this table as guidance to establish the GC temperature

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program and flow rate necessary to separate the analytes of interest.

GC Operating Conditions for PCBs as Aroclors

Carrier gas (He or H_2) flow rate	6 mL/minute (He)
Makeup gas (N_2) flow rate	50-100 mL/minute
Temperature program	150°C hold for 0 minute, 12°C/minute to 310°C, hold for 4.17 minutes
Injector temperature	230°C
Detector temperature	350°C
Injection volume	1 µL
Solvent	Hexane
Type of injector	Flash vaporization
Detector type	Dual ECD
Type of splitter	Restek press-fit Y-shaped splitter
These conditions are a recommended starting point. Each instrument may need adjustments for optimization. See instrument maintenance log for individual conditions.	

- Chromatographic conditions are adjusted to give adequate separation of the characteristic peaks in each Aroclor.
- Once established, the same operating conditions must be used for the analysis of samples and standards.

6.2 Supplies

- Volumetric flasks - 10 mL and 25 mL, for preparation of standards, Class A.
- Sodium sulfate is used for blank and LCS soil matrix.

7.0 Reagents and Standards

7.1 Reagent water, analyte-free, ASTM Type II or equivalent.

7.2 Reagent grade or pesticide grade chemicals are used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

7.3 n-Hexane, C_6H_{14}, (commercial): pesticide grade or equivalent.

7.4 Acetone, $(CH_3)_2CO$, commercial: pesticide quality or equivalent.

7.5 Primary Stock Standard Solutions (1000 mg/L): Purchased as certified solutions (Ultra, Supelco, Restek or equivalent). Commercially prepared stock standard solutions may be used at any concentration if they are certified by the manufacturer or by an independent source. Currently available standards are:

Aroclor	Supelco Catalog No.
1221	4-8098
1016	4-8097
1232	4-4805
1242	4-4806
1248	4-4807
1254	4-4808
1260	4-4809
1262*	4-4810

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Aroclor	Supelco Catalog No.
1268*	5-02146

Equivalent substitutes are acceptable.
*By request only.

- **Primary Working Stock Standard**: Add 100 µL of 1000 µg/mL standard to 10 mL hexane for a final concentration of 10 µg/mL. (Add surrogate as described in the next section.)

7.6 Surrogate standards: When PCBs are to be determined as Aroclors, tetrachloro-meta-xylene (TCMX) and decachlorobiphenyl (DCB) are used as surrogates and are added to each sample **prior** to extraction. Purchase Ultra ISM-320, or equivalent, at 200 µg/mL.

- Working standard for calibration standards and CCV: Add 50 µL of surrogate to above **Primary Working Stock Standard** for a final concentration of 1 µg/mL surrogate.
- Working standard for extracted samples and QC: Dilute 2.5 mL of TCMX/DCB standard at 200 µg/mL to a final volume of 1000 mL (0.5 µg/mL).

7.7 Secondary Stock Standards for **ICV, LCS and Spikes:** Vary which Aroclor is used for LCS and MS spiking, so that each target Aroclor is used within a two-year period. The spiking mix must be from a different source than the calibration standards. Currently available standards (1000 µg/mL each) are:

Aroclor	Restek Catalog No.
1016/1260	32039
1242	32009
1248	32010
1254	32011
1221	32007
1232	32008

Equivalent substitutes are acceptable.

- For the working secondary standards for the ICV, prepare separate 10 µg/mL working standards for each Aroclor by diluting 100 µL of the Aroclor at 1000 µg/mL to a final volume of 10 mL in hexane. Prepare Aroclor 1016 and 1260 together.
- For the working secondary standard for the LCS and MS/MSD, dilute 1.0 mL of the 1000 µg/mL Aroclor to a final volume of 200 mL with acetone in a Class A volumetric for a concentration of 5 µg/mL secondary working standard.

7.8 DDT Analog Standard: A standard containing DDT, DDE, and DDD is used to determine if these compounds elute at the same retention times as any of the target analytes (congeners, Aroclors). Purchase Ultra Scientific, Cat. No. PPM-828-1, 250 µg/mL DDT, DDE, DDD in isooctane, or equivalent.

7.9 Store stock standards and opened standards at -10 to -20°C; store standard solutions and surrogate solutions at approximately 6°C in the dark. All opened standards are good for 6 months. Transfer the stock standard solutions into Teflon™-sealed, screw-cap bottles. Check frequently for signs of degradation or evaporation, especially just prior to preparing calibration standards from them. Store surrogate spiking solutions in PTFE-sealed containers. Additional standard storage information is found in SOP Reagent and Standard Purchase, Preparation, Control, Documentation / NV08-214.

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8.0 Sample Collection, Preservation, Shipment and Storage

Matrix	Sample Container	Min. Sample Size	Preservation	Holding Time	Reference
Water	1-L, amber glass, fitted with a screw cap lined with Teflon™	500 mL	Cool 0-6°C. If amber bottles are not available, protect samples from light.	From collection to extraction: 1 year (CT, MA, AZ from collection to extraction: 7 days); 40 days from extraction to analysis	SW-846 Chapter 2
Soil, Oil	4-oz. amber-glass, Teflon™-lined cap	30 g	Cool 0-6°C. If amber bottles are not available, protect samples from light.	From collection to extraction: 1 year (CT, MA, AZ from collection to extraction: 14 days); 40 days from extraction to analysis	

The bottle and cap liner must be washed, rinsed with acetone or methylene chloride, and dried before use to minimize contamination. Certified clean bottles are acceptable. Chemical preservation is not used.

9.0 Quality Control

The laboratory maintains a formal quality assurance program and records to document the quality of the data generated. See Section 11 for QC equations.

Certain quality control and reporting criteria may vary depending on whether SW-846 8000B or 8000C criteria are required. In these cases, both sets of criteria have been noted in this SOP. 8000C criteria are required to be applied ONLY to Arizona and Washington samples. All other samples must be processed against referenced 8000B criteria. Exceptions may be required on a project-specific basis. See Section 1.7.

9.1 Sample QC

The following quality control samples are prepared with **each batch of no more than 20 samples, except for samples from OK and WY where a batch is no more than 10 samples.**			
QC Check	**Frequency**	**Acceptance Criteria**	**Corrective Action[1]**
Method blank	One per analytical batch, not to exceed 20 samples.	No analytes detected ≥ ½ RL or MDL, whichever is greater	Correct problem then re-prep and analyze method blank and all samples processed with the contaminated blank.
LCS[3] for all analytes, must be from a 2nd source.	One[3] per prep batch, not to exceed 20 samples.	See Control Limits Manual	Re-prep[2] and analyze the LCS and all samples in the affected analytical batch. If analyte is ND and LCS high, OK to report.
MS/MSD, must be from a 2nd source.	One per batch per matrix; if insufficient sample for MS/MSD, then data is qualified to indicate insufficient sample for MS/MSD.	See Control Limits Manual	None (LCS is used to determine if data are acceptable).
Surrogate(s)	Every sample, spike, standard, and method blank	See Control Limits Manual	Check system, re-inject, re-extract[2]. If analyte is ND and surrogate high, OK to report.

[1]All abnormalities must be noted on the data, the benchsheet, and in LIMS.

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[2]If unable to re-extract the samples because of insufficient sample volume or holding time has expired, then place a comment on the data, the benchsheet and in LIMS.
[3]For AZ, TX, WV samples, a LCS duplicate is required.

- **Method blank:** For each batch of samples extracted, a method blank is extracted and analyzed for the compounds of interest. The blank (reagent water for water batches, sodium sulfate for soil batches) is carried through all stages of the sample preparation and measurement.
  - If the method blank is detected for an analyte ≥ ½ RL or MDL, whichever is greater, it must be reanalyzed. If this fails to correct the problem, all associated samples with concentrations above the criteria for that analyte must be re-extracted. However, if the associated samples are non-detect, then reanalysis of the Method Blank is not required and re-extraction of samples is not required.
- A **Laboratory Control Sample (LCS)** is analyzed with each batch. Reagent water or sodium sulfate is spiked with a known amount of a **second-source standard** and extracted in the same way as the other samples.
  - 1.0 mL of the 5 µg/mL working.standard is added to the LCS (500 mL reagent water or 30 g sodium sulfate) prior to extraction. Concentration is 2.5 µg/mL for a 2 mL extract for waters and 0.5 µg/mL for a 10 mL extract for soils.
  - If the LCS fails, all samples extracted with the failed LCS must be re-extracted and re-analyzed. If the LCS recovery is high, and the associated samples are < RL, corrective action is not necessary, but data must be flagged.
- A **Matrix Spike / Matrix Spike duplicate pair** is analyzed with each batch of 20 or fewer samples. Two identical portions of a client's sample are spiked with a known amount of the second-source standard and extracted according to the procedure. Accuracy and precision are evaluated against control limits in the CLM to determine if there is a matrix effect on the analysis.
  - 1.0 mL of the 5 µg/mL working.standard is added to spike samples (500 mL or 30 g) prior to extraction. Concentration is 2.5 µg/mL for a 2 mL extract for waters and 0.5 µg/mL for a 10 mL extract for soils.
  - Analyze spikes to determine the concentration after spiking of each parameter. Calculate each percent recovery. Compare the percent recovery for each parameter with the corresponding QC acceptance criteria found in the CLM.
  - If any individual % recovery falls outside the designated range for recovery, that parameter has failed the acceptance criteria. Evaluate the LCS for batch acceptance.
- Known amounts of **surrogate standards** are added to all QC samples and client samples **prior** to extraction to determine if recovery is within the control limits in the CLM.
  - 1.0 mL of the working surrogate standard for extracted samples and QC is added to each sample (500 mL or 30 g) prior to extraction. Extract concentration is 0.1 µg/mL for a 2 mL extract.
  - If the surrogate recovery in the client sample is outside control limits, re-analyze and, if sufficient sample is available, re-extract and re-run. Place a comment in LIMS if it confirms. If the sample target(s) is ND and the surrogate is high, data is acceptable.
  - If the surrogate % recovery exceeds the upper control limit in a sample AND the sample target is greater than the RL, re-prep and re-analyze the batch.

9.2 Instrument QC

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QC Check	Frequency	Acceptance Criteria	Corrective Action[1]
Minimum five-point initial calibration for 1016/1260; single-point standards for remaining Aroclors[2]	Initial calibration prior to sample analysis. Perform instrument re-calibration once per year minimum or if ICV or CCV fails.	Linear regression correlation coefficient $r^2 \geq 0.99$, $r \geq 0.995$. RSD of CF ≤ 20%	Correct problem then repeat initial calibration
Initial calibration verification (ICV) must be from a 2nd source	Once immediately following initial calibration	All Aroclors within 15% of expected value for 8000B (±20% for 8000C)	Correct problem then repeat initial calibration
Initial Calibration Blank (ICB)	After ICV	< ½ RL or MDL, whichever is greater.	Prepare another blank. If same response, determine cause of contamination.
Continuing calibration verification (CCV)	Before sample analysis, after every 10 samples (or every 12 hours), and at the end of the analysis sequence	All analytes within 15% of expected value for 8000B (±20% for 8000C) and within the RT Window	Correct problem then repeat CCV (re-calibrate if necessary) and re-analyze all samples since last successful CCV. If high and samples ND, OK to report.
CCB	After each CCV	< ½ RL or MDL, whichever is greater.	Prepare another blank. If same response, determine cause of contamination, correct and re-run affected samples.
Second-column confirmation	100% for all positive results	Same as for initial or primary column analysis	Same as for initial or primary column analysis. If the relative % difference of results between the two columns is greater than 40%, a comment is placed in LIMS. Report the higher value for 8000B, the lower value for 8000C.
Retention time window calculated for each analyte.	System set-up, with each new column or major instrument maintenance. Update the mid-RTW at the start of the run or daily.	Each analyte of the LCS, MS/MSD and CCV must be within the calculated RTW.	Correct the problem and re-process or re-analyze samples.
DDT Analog Standard	Same time as Retention time window determination for each analyte.	Confirm that no calibration congener co-elutes with DDD, DDE, or DDT.	Adjust the GC conditions to achieve better resolution, or choose another peak that is characteristic of that Aroclor and does not correspond to a peak from a DDT analog.
MDL Verification (extracted)	Minimum yearly	Detectible	Re-evaluate MDL standard used and MDL; see Technical Director.

[1]All abnormalities must be noted on the data, the benchsheet, and in LIMS.
[2]Single-point standards for other Aroclors, unless linear regression is used, then each Aroclor must be calibrated by linear regression.

- Calibration is detailed in Section 10.
- **Second-Source Independent Calibration Verification (ICV):** Separate ICV standards for each Aroclor are run immediately after the initial calibration. Aroclors 1016 and 1260 are prepared together. Agreement must be ± 15% for 8000B (± 20% for 8000C); if not, re-calibrate.
  - Add 100 µL of the working secondary standard to 1.0 mL for 1.0 µg/mL standard.
- The **Initial Calibration Blank's (ICB's)** purpose is to assess contamination of the instrument. Run the ICB after the ICV (results < ½ RL or MDL, whichever is greater).

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- **Continuing Calibration Verification (CCV):** The working calibration curve, or calibration factor, is verified on each working day by the measurement of one or more calibration standards (**CCV**) every 10 samples (or every 12 hours) and at the end of the analysis.
  - The CCV is a mixture of Aroclor 1016 and Aroclor 1260 prepared at 1.0 µg/mL (100 µL of a 10 µg/mL Working Stock Standard is diluted to 1000 µL hexane). Also, each sequence, the most commonly reported Aroclors (1242, 1254, 1248) are confirmed with a 1.0 µg/mL standard If an Aroclor other than 1016/1260 is detected and the 1.0 µg/mL verification did not meet the requirements, the sample is rerun after a single-point calibration of the detected Aroclor. If an Aroclor is detected within a sequence that has not been confirmed (i. e., 1221, 1232, 1262, 1268), the sample is rerun after a 1.0 µg/mL CCV of the detected Aroclor and compared to the above requirements.
    - Required acceptance is ± 15% for 8000B (± 20% for 8000C). When a CCV fails to meet the QC criteria:
      - All samples that were injected after the last CCV that met the QC criteria must be re-analyzed. All samples must be bracketed by acceptable CCVs.
      - However, if the CCV exhibits a response for an analyte that is <u>above</u> the acceptance limit and the analyte was <u>not</u> detected in the specific samples analyzed during the analytical shift, then the extracts for those samples do not need to be reanalyzed and may be reported with qualifiers.
      - Repeat the failed CCV by injecting another aliquot of the CCV. If the response is still not within acceptance criteria, then a new initial calibration must be prepared.
- The **Continuing Calibration Blank (CCB),** run after each CCV, must be < ½ RL or MDL, whichever is greater; if not, prepare a second blank. If the same response, determine the source of the contamination and re-run the affected samples.
- **Second column confirmation:** All the criteria for the primary column must be met.
- **Retention Time Windows:** .Retention time windows are crucial to the identification of target compounds. Absolute retention times are used for the identification of the Aroclors. Retention time windows are established to compensate for minor shifts in absolute retention times as a result of sample loadings and normal chromatographic variability. The width of the retention time window is carefully established to minimize the occurrence of both false positive and false negative results. Tight retention time windows may result in false negatives and/or may cause unnecessary reanalysis of samples when surrogates or spiked compounds are erroneously not identified. Overly wide retention time windows may result in false positive results that cannot be confirmed upon further analysis.
  - Typical retention times are:

Compound	Rtx-5 RT (minute)	Rtx-50 RT (minute)
Aroclor 1016	5.648	7.03
Aroclor 1221	5.06	6.41
Aroclor 1232	7.22	7.79
Aroclor 1242	7.221	8.72
Aroclor 1248	8.134	9.75
Aroclor 1254	8.951	10.34
Aroclor 1260	9.898	12.37
Aroclor 1262	9.90	11.61
Aroclor 1268	12.249	13.74
mp = multi-peak		

  - Confirm that common single-component pesticides, such as DDT, DDD, and DDE, do not elute at the same retention time as the congeners used for calibration by running the **DDT**

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Analog Standard at the same time as the determination of retention time windows. If, during Aroclor analysis, any of the DDT analogs elute at the same retention time as an Aroclor peak that was chosen for use in quantitation, then adjust the GC conditions to achieve better resolution, or choose another peak that is characteristic of that Aroclor and does not correspond to a peak from a DDT analog.

- **Method Detection Limit Verification**: Annually, at a minimum, with an **extracted** low standard, verify that the MDL is detectible; if not, re-evaluate the MDL.

10.0 Procedure

10.1 Calibration: Refer to SOP Selection of Calibration Points / CA-T-P-002. See Section 11 for equations. Calculations are performed by vendor software and LIMS.

See Section 11 for equations.

1 Using the external standard calibration procedure, prepare the following calibration standards:

Amount of Working Stock Std (µL)	Final Volume (mL)	Conc. (µg/mL)	Surrogate Concentration (µg/mL)
3.0	1.0	0.03	0.003
5.0	1.0	0.05	0.005
20	1.0	0.20	0.02
50	1.0	0.5	0.05
100*	1.0	1.0*	0.1*
200	1.0	2.0	0.2
300	1.0	3.0	0.3

Prepare the Working Stock Standard as described in Section 7: 10 µg/mL Aroclor and 1 µg/mL surrogate. *CCV also.

A standard containing a mixture of 1:1 Aroclor 1016 and Aroclor 1260 includes many of the peaks represented in the other Aroclor mixtures. As a result, a multi-point initial calibration employing a mixture of Aroclors 1016 and 1260 at seven concentrations is sufficient to demonstrate the linearity of the detector response without the necessity of performing initial calibrations for each of the Aroclors. This standard is also used to determine the concentrations of either Aroclor 1016 or Aroclor 1260, if they are present in a sample. Prepare calibration standards containing equal concentrations of both Aroclor 1016 and Aroclor 1260 by dilution of the Working Stock Standard with hexane. The concentrations define the linear range of the detector. **One standard must be at or below the RL.**

2 Single standards of each of the other seven (including 1262 and 1268, if requested by client) Aroclors are required to aid the analyst in pattern recognition. These single standards (at 1 µg/mL) of the remaining Aroclors are then used to determine a single point calibration factor for each respective Aroclor unless linear regression is used. The standards for these Aroclors are analyzed **before** the analysis of any samples and may be analyzed before or after the analysis of the five 1016/1260 standards. **The calibration model used for 1016/1260 must also be used for each individual Aroclor.**

3 A 1 µL injection of each calibration standard is used for calibration (and sample analysis).

4 Record the peak area for each characteristic Aroclor peak to be used for quantitation. A minimum of 3 peaks must be chosen for each Aroclor, and preferably 5 peaks. The peaks must be characteristic of the Aroclor in question. Choose peaks in the Aroclor standards that are at least 25% of the height of the largest Aroclor peak. For each Aroclor, the set of 3 to 5

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	peaks must include at least one peak that is unique to that Aroclor. Use at least five peaks for the Aroclor 1016/1260 mixture, none of which are found in both of these Aroclors.
5	Calculate the calibration factor (CF) for each characteristic Aroclor peak in each of the initial calibration standards. Five sets of calibration factors are generated for the Aroclor 1016/1260 mixture, each set consisting of the calibration factors for each of the five peaks chosen for this mixture. The single standard for each of the other Aroclors generates at least three calibration factors, one for each selected peak.
6	The calibration factors from the initial calibration are used to evaluate the linearity of the initial calibration. If the RSD of the calibration factors is ≤ 20% over the calibration range, then linearity through the origin is assumed, and the average calibration factor is used to determine sample concentrations. If the RSD is greater than 20%, then first-order, linear regression is used. The correlation coefficient (r^2) must be greater than or equal to 0.99 (or r ≥ 0.995). If the curve is not linear by either method, then identify and fix the problem and re-calibrate. **Higher order equations are not allowed.**
7	When the Aroclor 1016/1260 mixture is used to demonstrate the detector response, the calibration model chosen for any of the five peaks in 1016 or 1260 **must** be applied to the other peaks of that Aroclor.
8	Evaluate the ICV and ICB.
9	Calibration Sequence
10	Both columns must meet all calibration acceptance criteria.

1	Instrument Blank
2	Initial Calibration, if required
3	ICV (1016, 1260)
4	ICV (1242)
5	ICV (1254)
6	ICV (1248)
7	ICV (1232)
8	ICV (1221)
9	ICB

10.2 Gas Chromatography

1	**The same GC operating conditions used for the initial calibration must be employed for sample analyses.**
2	At the beginning of the sequence, center peaks on the window. If any of the standards fall outside their daily retention time windows, the system is out of control. Determine the cause of the problem and correct it. Rerun affected samples.
3	If the responses in the sample chromatogram exceed the upper calibration standard, dilute the extract with a known amount of hexane and reanalyze.
4	Sample injections may continue for as long as the calibration verification standards meet instrument QC requirements. The sequence ends when the set of samples has been injected or when qualitative or quantitative QC criteria are exceeded.

10.3 Recommended Sample Analysis Sequence:

1	Instrument Blank
2	CCV (1016, 1260)

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3	CCV (1242)
4	CCV (1254)
5	CCV (1248)
6	CCV (1232)
7	CCV (1221)
8	CCB
9	Method Blank
10	Matrix Spike
11	Matrix Spike Duplicate
12	LCS
13	Samples 1- 10 or every 12 hours
14	CCV (1016, 1260)
15	CCB
16	Samples 11- 20 or every 12 hours
17	CCV (1016, 1260)
18	CCB

If compound identification or quantitation is precluded due to interference (e. g., broad, rounded peaks or ill-defined baselines are present), cleanup of the extract or replacement of the capillary column, or detector is performed.

10.4 Qualitative identification - The identification of PCBs as Aroclors using this method with an electron capture detector is based on agreement between the retention times of peaks in the sample chromatogram with the retention time windows established through the analysis of standards

- Tentative identification of an analyte occurs when a peak from a sample extract falls within the established retention time window for a specific target analyte. Each tentative identification must be confirmed: using a second GC column of dissimilar stationary phase.
- When simultaneous analyses are performed from a single injection (using the dual-column GC configuration, it is not practical to designate one column as the analytical (primary) column and the other as the confirmation column). Since the calibration standards are analyzed on both columns, the results for both columns must meet the calibration acceptance criteria. If the retention times of the peaks on both columns fall within the retention time windows on the respective columns, then the target analyte identification has been confirmed.

10.5 Quantitation is accomplished by comparison of the sample chromatogram to that of the most similar Aroclor standard.

- Use the individual Aroclor standards (not the 1016/1260 mixtures) to determine the pattern of peaks on Aroclors 1221, 1232, 1242, 1248, 1254, and, if requested, 1262 and 1268. The patterns for Aroclors 1016 and 1260 are evident in the mixed calibration standards.
- Once the Aroclor pattern has been identified, compare the responses of 3 to 5 major peaks in the single-point calibration standard for that Aroclor with the peaks observed in the sample extract. The amount of Aroclor is calculated using the individual calibration factor for each of the 3 to 5 characteristic peaks and the calibration model established from the multi-point calibration of the 1016/1260 mixture. A concentration is determined using each of the characteristic peaks and then those 3 to 5 concentrations are summed to determine the concentration of that Aroclor. If the instrument linearity has been demonstrated using linear regression for the Aroclor 1016/1260 standard, the CF from the single point Aroclor standard is not used. Each Aroclor then requires multi-point calibration.
- Calculate results from both columns and report the **higher** of the two values (if using **8000B)**

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or the **lower** (if using **8000C**) if no visible interference is observed. If the results differ by more than 40%, add a comment in LIMS.

- Weathering of PCBs in the environment and changes resulting from waste treatment processes may alter the pattern of a specific Aroclor so that it is no longer recognizable. Samples containing more than one Aroclor present similar problems.

10.6 Chromatographic System Maintenance: When system performance does not meet the established QC requirements, maintenance is required and may include one or more of the following: With regard to splitter connections, for dual columns which are connected using a Y-shaped fused-silica connector, replace with a new deactivated splitter. Cut off the first few inches (up to one foot) of the injection port side of the pre-column.

11.0 Calculations / Data Reduction

11.1 Accuracy

% Recovery = Measured concentration x 100
Known concentration

11.2 Precision (RPD)

RPD = Absolute value (orig. sample value - dup. sample value) x 100
(Orig. sample value + dup. sample value)/2

11.3 Calibration Factor

Calibration Factor = Peak areas of standard
Concentration of the standard

11.4 Standard Deviation, Relative Standard Deviation

%RSD = (SD / $\overline{x_i}$) X 100

SD = Standard Deviation of initial 5 CFs for each compound calculated as follows:

$$SD = \sqrt{\sum_{i=1}^{n} \frac{\left(x_i - \overline{x_i}\right)^2}{n-1}}$$

$\overline{x_i}$ = Mean (Average) of initial 5 CFs for each compound.

n = number of standards

x_i = individual CF

11.5 Linear Calibration Using a Least Squares Regression: A linear calibration model based on a least squares regression is employed for Aroclors that do not meet the RSD limits. For external standard calibration, x is the mass of the analyte in the sample aliquot introduced into the instrument and y is the area (or height) or the response, as in:

$x = C_s$ and $y = A_s$

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A linear least squares regression attempts to construct a linear equation of the form:

$$y = ax + b$$

The mathematics used in least squares regression has a tendency to favor numbers of larger value over numbers of smaller value. Thus the regression curves generated tend to fit points at the upper calibration levels better than points at the lower calibration levels. To compensate for this, a weighting factor which reduces this tendency is used. Examples of acceptable weighting factors which place more emphasis on numbers of smaller value are:

$$w = 1/y \quad \text{or} \quad w = 1/y^2$$

Do not include the origin (0, 0) as an extra calibration point. Re-process each calibration standard as an unknown to determine the best fit model. Each calibration point above the RL must be ± 15% true (8000B) or ±20% true (8000C); the RL-level standard must be ± 30% true.

The regression calculation generates a correlation coefficient (r) that is a measure of the "goodness of fit" of the regression line to the data. A value of 1.00 indicates a perfect fit. In order to be used for quantitative purposes, r must be greater than or equal to 0.995 or $r^2 \geq 0.99$.

11.6 Coefficient of Determination **Correlation Coefficient**

$$r^2 = \frac{\left(\sum xy\right)^2}{\sum x^2 \sum y^2} \qquad r = \frac{\left(\sum xy\right)}{\sqrt{\sum x^2 \sum y^2}}$$

y = Response
x = Concentration

11.7 % Difference, % Drift

% Difference = $\frac{(CF_v) - (\text{Avg. CF})}{\text{Avg. CF}} \times 100$

CF_v = CF from verification standard
Avg. CF = Average CF from Initial Calibration.

% Drift = $\frac{\text{Result} - \text{True Value}}{\text{True Value}} \times 100$

11.8 Concentration Calculations:

- For **aqueous** samples:

Conc. (µg/L) = $\frac{A_x V_t D(1000)}{CF_{mean} V_s} = \frac{(\mu g/mL \text{ instrument})(D)(\text{extract volume in mL})(1000)}{\text{mL sample extracted}}$

A_x = Area of the analyte in the sample.
V_t = Total volume of the concentrated extract (mL).

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D = Dilution factor, if the sample or extract was diluted prior to analysis. If no dilution was made, D = 1. The dilution factor is always dimensionless.
CF_{mean} = Mean calibration factor from the initial calibration (area/concentration).
V_s = Volume of the aqueous sample extracted in mL.

- For **non-aqueous** samples:

$$\textbf{Conc. (µg/kg)} = \frac{A_x V_t D\,(1000)}{CF_{mean}\, W_s} = \frac{(\mu g/mL \text{ instrument})(D)(\text{extract volume in mL})(1000)}{\text{g sample extracted}}$$

A_x, V_t, D, CF_{mean} are the same as for aqueous samples, and
W_s = Weight of sample extracted (g). The wet weight or dry weight may be used, depending upon the specific application of the data.

12.0 Method Performance

12.1 Method Detection Limit Study (MDL): The method detection limit (MDL) is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. The MDL is determined according to the laboratory's MDL procedure in SOP Determination of Method Detection Limits / NV08-202. MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix, and may not be achievable in all environmental matrices. The laboratory maintains MDL studies for analyses performed; these are verified at least annually unless method requirements require a greater frequency.

12.2 Demonstration of Capability: The laboratory demonstrates initial proficiency by generating data of acceptable accuracy and precision for target analyses in a clean matrix. The laboratory also repeats the operation whenever new staff is trained or significant changes in instrumentation are made and on an annual basis thereafter. See the training section of TestAmerica Nashville's QA Manual and SOP Training / NV08-199 for information on how to accomplish this demonstration.

12.3 Training Requirements: Demonstration of Capability is performed initially when learning the method and annually thereafter. Four Laboratory Control Samples resulting in an average % recovery within the control limits and a precision less than the quality control maximum are required for each matrix analyzed.

12.4 Proficiency Testing Studies: The laboratory participates in formal proficiency testing (PT) studies, where solutions of unknown concentrations are analyzed and the performance of all participants is compared. See the QA department for the results of recent PT studies.

13.0 Pollution Control

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i. e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention.

14.0 Waste Management

14.1 Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes must be stored, managed, and disposed of in accordance with all federal and state laws and regulations. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to the QA Manual and SOP Waste Disposal / NV10-83.

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14.2 Wastestreams Produced by the Method:

- Extracted samples are discharged into the sanitary sewer or trash receptacle.
- Halogenated solvents are taken to waste disposal and placed in the halogenated waste drum.
- PCB oils are taken to waste disposal and placed in the PCB oil separatory funnel.

15.0 References / Cross-References

15.1 Method 8082, SW-846 Update III, Revision 0, December 1996, and 8082A, Update IV, Revision 1, February 2007.

15.2 Method 8000B, SW-846, Revision 2, December 1996 and Method 8000C, Revision 3, March 2003.

15.3 TestAmerica Nashville's Quality Assurance Manual and Control Limits Manual.

15.4 Corporate Environmental Health and Safety Manual (CW-E-M-001)

15.5 SOPs: Selection of Calibration Points / CA-T-P-002, Manual Integration / CP0101-03, 3510 / NV03-24, 3550 / NV03-23, 3541 / NV03-231, 3580 / NV03-106, 3660 /NV03-99, 3665 /NV03-117, 8270 / NV04-22, CLP4.2-Pest / NV04-129, Waste Disposal / NV10-83, Training Procedures for Environmental Technical Staff / NV08-199, MDL / SA08-202, Reagent and Standard Purchase / NV08-214.

15.6 Controlled Document: QAF-45, TestAmerica Nashville – Acronyms, Keywords, and Definitions.

16.0 Method Modifications

None.

17.0 Attachments

None.

18.0 Revision History

- Revision 10, dated 30 September 2008
  - Integration for TestAmerica and STL operations.
  - Inclusion of corrective action and equation information from QA Manual.
  - Additional detail and update on standards make-up.
  - Incorporated 8082A requirements.
- Revision 11, dated 30 October 2009
  - General editing for redundancies and clarity.
  - Incorporate amendment 10a (holding time change).
  - Corporate review.
- Revision 12, dated November 30 2010
  - Addition of QAF-45 and Section 14.2.
  - Addition of the oil matrix instructions.
  - Replace ICAL with Initial calibration, if required.
  - Addition of AZ to list of states with short holding times.

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Title: DETERMINATION OF VOLATILE ORGANICS BY GC/MS BASED ON METHODS 8260B AND 8260A

[Method: EPA Methods 8260B AND 8260A]

Approvals (Signature/Date):

Thomas E. Stiller 01/02/09
Technology Specialist Date

1/02/09
Health & Safety Coordinator Date

Dorothy J. Leeson 1/2/09
Quality Assurance Manager Date

Mark L. Bruce 1/2/09
Technical Director Date

1/5/09
Laboratory Director Date

This SOP was previously identified as SOP No. NC-MS-019, Rev 0, dated 06/30/08

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1.0 SCOPE AND APPLICATION

1.1. This method is applicable to the determination of Volatile Organic Compounds in waters, wastewater, soils, sludges, and other solid matrices. Standard analytes are listed in Tables 5 and 6.

1.2.1. This SOP is applicable to Method 8260B. It may also be used for analysis following Method 8260A. The associated LIMS method codes are QK (8260B) and MZ (8260A). Ohio VAP projects are distinguished by Program Code 2J. The following Prep Codes are used: 15 (5 mL purge), 25 (low-level 5mL purge), 4B (5035, Methanol Preservation, EnCore™), 4D (5035, Sodium Bisulfate Preservation, EnCore™), 4P (Frozen, EnCore™), M8 (5035A, Frozen Encore™), and 73 (5030A Methanol Prep).

1.3. This method can be used to quantify most volatile organic compounds that have boiling points below 200°C and are insoluble or slightly soluble in water. Volatile water-soluble compounds can be included in this analytical technique; however, for more soluble compounds, quantitation limits are approximately ten times higher because of poor purging efficiency.

1.4. The method is based upon a purge and trap, gas chromatograph/mass spectrometric (GC/MS) procedure. The approximate working range is 5 to 200 µg/L for 5 mL waters, 1 to 40 µg/L for low-level waters, 5 to 200 µg/kg for low-level soils, and 250 to 10,000 µg/kg for medium-level soils. Reporting limits are listed in Tables 1 and 3.

1.5. Method performance is monitored through the use of surrogate compounds, matrix spike/matrix spike duplicates, and laboratory control spike samples.

2.0 SUMMARY OF METHOD

2.1. Volatile compounds are introduced into the gas chromatograph by the purge and trap method. The components are separated via the chromatograph and detected using a mass spectrometer, which is used to provide both qualitative and quantitative information.

2.2. Aqueous samples are purged directly. Soils are preserved by extracting the volatile analytes into methanol. Soil samples may be preserved with sodium bisulfate or by freezing and purging directly.

2.3. In the purge and trap process, an inert gas is bubbled through the solution at ambient temperature or at 40°C (40°C required for low level soils) and the volatile components are efficiently transferred from the aqueous phase to the vapor phase. The vapor is swept through a sorbant column where the volatile components are trapped. After purging is completed, the sorbant column (trap) is heated and backflushed with inert gas to desorb the components onto a gas chromatographic column. The gas chromatographic column is then heated to elute the components, which are detected with a mass spectrometer.

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2.4. Qualitative identifications are confirmed by analyzing standards under the same conditions used for samples and comparing the resultant mass spectra and GC retention times. Each identified component is quantified by relating the MS response for an appropriate selected ion produced by that compound to the MS response for another ion produced by an internal standard.

3.0 DEFINITIONS

3.1. Batch

The batch is a set of up to 20 samples of the same matrix processed using the same procedures and reagents within the same time period. Using this method, each BFB analysis will start a new batch. Batches for medium level soils are defined at the sample preparation stage and may be analyzed on multiple instruments over multiple days, although reasonable effort must be made to keep the samples together.

3.1.1. The Quality Control batch must contain a matrix spike/spike duplicate (MS/MSD), a Laboratory Control Sample (LCS), and a method blank. Refer to the TestAmerica QC program document (QA-003) for further details of the batch definition.

3.2. Method Blank

3.2.1. A method blank consisting of all reagents added to the samples must be analyzed with each batch of samples. The method blank is used to identify any background interference or contamination of the analytical system which may lead to the reporting of elevated concentration levels or false positive data.

3.3. Laboratory Control Sample (LCS)

3.3.1. Laboratory Control Samples are well characterized, laboratory generated samples used to monitor the laboratory's day-to-day performance of routine analytical methods. The LCS, spiked with a group of target compounds representative of the method analytes, is used to monitor the accuracy of the analytical process, independent of matrix effects. Ongoing monitoring of the LCS results provides evidence that the laboratory is performing the method within accepted QC guidelines for accuracy and precision.

3.4. Surrogates

3.4.1. Surrogates are organic compounds which are similar to the target analyte(s) in chemical composition and behavior in the analytical process, but which are not normally found in environmental samples. Each sample, blank, LCS, and MS/MSD is spiked with surrogate standards. Surrogate spike recoveries must be evaluated by determining whether the concentration (measured as percent recovery) falls within

the required recovery limits.

3.5. Matrix Spike/Matrix Spike Duplicate (MS/MSD)

3.5.1. A matrix spike is an environmental sample to which known concentrations of target analytes have been added. A matrix spike duplicate is a second aliquot of the same sample, which is prepared and analyzed along with the sample and matrix spike. Matrix spikes and duplicates are used to evaluate accuracy and precision in the actual sample matrix.

3.6. Calibration Check Compound (CCC)

3.6.1. CCCs are a representative group of compounds, which are used to evaluate initial calibrations and continuing calibrations. Relative percent difference for the initial calibration and % drift for the continuing calibration response factors are calculated and compared to the specified method criteria.

3.7. System Performance Check Compounds (SPCC)

3.7.1. SPCCs are compounds, which are sensitive to system performance problems and are used to evaluate system performance and sensitivity. A response factor from the continuing calibration is calculated for the SPCC compounds and compared to the specified method criteria.

4. INTERFERENCES

4.1. Method interferences may be caused by contaminants in solvents, reagents, glassware, and other processing apparatus that lead to discrete artifacts. All of these materials must be routinely demonstrated to be free from interferences under conditions of the analysis by running laboratory method blanks as described in the Quality Control section. All glassware is cleaned per SOP NC-QA-014. The use of ultra high purity gases, prepurged purified reagent water, and approved lots of purge and trap grade methanol will greatly reduce introduction of contaminants. In extreme cases, the purging vessels may be pre-purged to isolate the instrument from laboratory air contaminated by solvents used in other parts of the laboratory.

4.2. Samples can be contaminated by diffusion of volatile organics (particularly methylene chloride and fluorocarbons) into the sample through the septum seal during shipment and storage. A field blank prepared from reagent water and carried through the sampling and handling protocol can serve as a check on such contamination. Refer to SOP NC-QA-020 for additional information on holding blanks.

4.3. Matrix interferences may be caused by non-target contaminants that are co-extracted from

the sample. The extent of matrix interferences will vary considerably from source to source depending upon the nature and diversity of the site being sampled.

4.4. Cross-contamination can occur whenever high-level and low-level samples are analyzed sequentially or in the same purge position on an autosampler. Whenever an unusually concentrated sample is analyzed, it must be followed by one or more blanks to check for cross-contamination. The purge and trap system may require extensive bake-out and cleaning after a high-level sample.

4.5. Some samples may foam when purged due to surfactants present in the sample. When this kind of sample is encountered, the sample must be diluted.

5. SAFETY

5.1 Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual and this document.

5.2. Eye protection that protects against splash, laboratory coat, and appropriate gloves must be worn while samples, standards, solvents, and reagents are being handled. Disposable gloves that have been contaminated must be removed and discarded; other gloves must be cleaned immediately. Cut-resistant gloves MUST be worn when opening VOA vials and when doing any other task that presents a strong possibility of getting cut.

5.3. Primary Materials Used

5.3.1. The following is a list of the materials used in this method, which have a serious or significant hazard rating. NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table. A complete list of materials used in the method can be found in the Reagents and Standards section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure

Sodium bisulfate	Irritant	None	Causes mild to severe irritation to the eyes. Prolonged exposure may cause burn if not flushed with water. May cause mild irritation to skin. Prolonged exposure may cause burn if not flushed with water.
Hydrochloric Acid	Corrosive Poison	5 ppm-Ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Methanol	Flammable Poison Irritant	200 ppm-TWA	A slight irritant to the mucous membranes. Toxic effects exerted upon nervous system, particularly the optic nerve. Symptoms of overexposure may include headache, drowsiness and dizziness. Methyl alcohol is a defatting agent and may cause skin to become dry and cracked. Skin absorption can occur; symptoms may parallel inhalation exposure. Irritant to the eyes.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

5.4. It is recommended that analysts break up work tasks to avoid repetitive motion tasks, such as opening a large number of vials or containers in one time period.

5.5. Exposure to chemicals must be maintained **as low as reasonably achievable.** All samples with a sticker that reads "Caution/Use Hood!" **must** be opened in the hood. Contact the EH&S Coordinator if this is not possible. Solvent and waste containers must be kept closed unless transfers are being made. MS VOA samples may be prepared outside of the hood, unless it is known that concentrations are high.

5.6. The preparation of standards and reagents must be conducted in a fume hood with the sash closed as far as the operations will permit. MS VOA standards may be prepared outside of the hood due to low concentrations of analytes.

5.7. All work must be stopped in the event of a known or potential compromise to the health and safety of a TestAmerica associate. The situation must be reported **immediately** to the EH&S Coordinator and the laboratory Group Leader.

5.8. Laboratory personnel assigned to perform hazardous waste disposal procedures must have a working knowledge of the established procedures and practices outlined in the TestAmerica Corporate Environmental Health and Safety Manual. These employees must have training on the hazardous waste disposal practices initially upon assignment of these tasks, followed

by an annual refresher training.

5.9. Specific Safety Concerns or Requirements

5.9.1. The gas chromatograph and mass spectrometer contain zones that have elevated temperatures. The analyst needs to be aware of the locations of those zones, and must cool them to room temperature prior to working on them.

5.9.2. The mass spectrometer is under deep vacuum. The mass spectrometer must be brought to atmospheric pressure prior to working on the source.

5.9.3. There are areas of high voltage in both the gas chromatograph and the mass spectrometer. Depending on the type of work involved, either turn the power to the instrument off, or disconnect it from its source of power.

5.9.4. Sodium bisulfate creates Sulfuric Acid when mixed with water.

6. EQUIPMENT AND SUPPLIES

6.1. Microsyringes: 10 µL and larger

6.2. Syringe: 5, 25, or 50 mL glass with luerlok tip, if applicable to the purging device.

6.3. Balance: Analytical, capable of accurately weighing 0.0001g, and a top-loading balance capable of weighing 0.1g

6.4. Glassware:

6.4.1. Vials: 20 and 40 mL with screw caps and Teflon® liners.

6.4.2. Volumetric flasks: 10 mL and 100 mL, class A with ground-glass stoppers.

6.5. Spatula: Stainless steel.

6.6. Disposable pipettes: Pasteur, 5 ¾ in.

6.7. pH paper: Wide range, pH 0-14.

6.8. Gases:

6.8.1. Helium: Ultra high purity, gr. 5, 99.999%.

6.8.2. Nitrogen: Ultra high purity from cylinders or gas generators may be used as an alternative to helium for purge gas.

6.9. Purge and Trap Device: The purge and trap device consists of the sample purger, trap, and desorber.

6.9.1. Sample Purger: The recommended purging chamber is designed to accept 5 mL samples with a water column at least 3 cm deep. The purge gas must pass through the water column as finely divided bubbles, each with a diameter of less than 3 mm at the origin. The purge gas must be introduced no more than 5 mm from the base of the water column. Alternative sample purge devices may be used provided equivalent performance is demonstrated. Low-level soils are purged directly from a VOA vial.

6.9.2. Trap: A variety of traps may be used, depending on the target analytes required. One of the traps used is the Vocarb 3000 trap. Other traps such as the OI 10 may be used if the Quality Control criteria are met. Refer also to instrument operating manuals located within the laboratory.

6.9.3. Desorber: The desorber must be capable of rapidly heating the trap to at least 180°C. Many such devices are commercially available.

6.9.4. Sample Heater: A heater capable of maintaining the purge device at 40°C is necessary for low-level soil analysis.

6.10. Gas Chromatograph/Mass Spectrometer System:

6.10.1. Gas Chromatograph: The gas chromatograph (GC) system must be capable of temperature programming.

6.10.2. Gas Chromatographic Columns: Capillary columns are used. Some typical columns are listed below:

6.10.2.1. Column 1: 20m x 0.18 ID DB-624 with 1 µm film thickness.

6.10.2.2. Mass Spectrometer: The mass spectrometer must be capable of scanning 35-300 AMU every two seconds or less, using 70 volts electron energy in the electron impact mode and capable of producing a mass spectrum that meets the required criteria when 50 ng of 4-Bromofluorobenzene (BFB) are injected onto the gas chromatograph column inlet.

6.10.3. GC/MS Interface: In general direct introduction to the mass spectrometer is used but any interface that achieves all acceptance criteria may be used.

6.10.4. Data System: A computer system that allows the continuous acquisition and storage on machine-readable media of all mass spectra obtained throughout the

duration of the chromatographic program. The computer must have software that allows searching any GC/MS data file for ions of a specified mass and plotting such ion abundances versus time or scan number. This type of plot is defined as an Extracted Ion Current Profile (EICP). Software must also be available that allows integrating the abundances in any EICP between the specified time or scan-number limits. Also, for the non-target compounds, software must be available that allows for the comparison of sample spectra against reference library spectra. The NIST/EPA mass spectral library must be used as the reference library. The computer system must also be capable of backing up data for long-term off-line storage.

7. REAGENTS AND STANDARDS

7.1. Reagents

7.1.1. Methanol: Purge and Trap grade, high purity

7.1.2. Reagent Water: High purity water that meets the requirements for a method blank when analyzed (see Section 9.3). Reagent water may be purchased as commercial distilled water and prepared by purging with an inert gas overnight. Other methods of preparing reagent water are acceptable.

7.1.3. Hydrochloric Acid – (1:1 v/v): Reagent grade or equivalent

7.1.4. Sodium bisulfate: Reagent grade or equivalent

7.2. Standards

7.2.1. Calibration Standard

7.2.1.1. Stock Solutions: Stock solutions may be purchased as certified solutions from commercial sources or prepared from pure standard materials as appropriate. These standards are prepared in methanol and stored in Teflon®-sealed screw-cap bottles with minimal headspace at -10° to -20°C.

7.2.1.2. Working standards: A working solution containing the compounds of interest prepared from the stock solution(s) in methanol. These standards are stored in the freezer or as recommended by the manufacturer. Working standards are monitored by comparison to the initial calibration curve. If any of the calibration check compounds drift in response from the initial calibration by more than 20% then corrective action is necessary. This may include steps such as instrument maintenance, preparing a new calibration verification standard or tuning the instrument. If the corrective actions do not correct the problem then a new initial calibration must be performed.

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7.2.1.3. Aqueous Calibration Standards are prepared in reagent water using the secondary dilution standards. These aqueous standards must be prepared daily.

7.2.1.4. If stock or secondary dilution standards are purchased in sealed ampoules, they may be used up to the manufacturer's expiration date.

7.2.1.5. Additional information can be found in SOP NC-QA-017.

7.2.2. Internal Standards: Internal standards are added to all samples, standards, and blank analyses. Refer to Table 7 for internal standard components.

7.2.3. Surrogate Standards: Refer to Table 8 for surrogate standard components and spiking levels.

7.2.4. Laboratory Control Sample Spiking Solutions: Refer to Table 9 for LCS components and spiking levels.

7.2.5. Matrix Spiking Solutions: The matrix spike contains the same components as the LCS. Refer to Table 9.

7.2.6. Tuning Standard: A standard is made up that will deliver 50 ng on column upon injection. A recommended concentration of 50 ng/µL of 4-Bromofluorobenzene in methanol is prepared as described in Sections 7.2.1.1 and 7.2.1.2.

7.2.7. All standard preparation information is detailed in the Standard Logbook.

8. SAMPLE COLLECTION, PRESERVATION, AND STORAGE

8.1. Holding times for all volatile analysis are 14 days from sample collection to analysis.

8.2. For DoD samples, water samples are normally preserved at pH $\leq$ 2 with 1:1 hydrochloric acid. Unpreserved water samples must be analyzed within seven days of sampling.

8.3. Solid samples are field preserved with sodium bisulfate solution or by freezing upon receipt at the laboratory for low-level analysis, or with methanol for medium-level analysis. Soil samples can also be taken using the EnCore™ sampler and preserved in the lab within 48 hours of sampling. Analysis must be completed 14 days from sampling. At specific client request, unpreserved soil samples may be accepted.

8.4. There are several methods of sampling soil. The recommended method, which provides the minimum of field difficulties, is to take an EnCore™ sample. (The 5g or 25g sampler can be used, depending on client preference). Following shipment back to the lab, the soil is preserved in methanol. This is the medium level procedure. If very low detection limits are needed (< 50 µg/kg for most analytes), then it will be necessary to use two additional 5g

EnCore™ samplers or to use field preservation.

8.5. Sample collection for medium level analysis using EnCore™ samplers.

8.5.1. Ship one 5g (or 25g) EnCore™ sampler per field sample position.

8.5.2. An additional 2 oz plastic bottle must be shipped for percent moisture determination.

8.5.3. When the samples are returned to the lab, extrude the (nominal) 5g (or 25g) sample into a tared VOA vial containing 5 mL methanol (25 mL methanol for the 25g sampler). Obtain the weight of the soil added to the vial and note on the label.

8.5.4. Add the correct amount of surrogate spiking mixture. (Add 25 µL of 2500 µg/mL solution for a nominal 25g sample, 5 µL for a nominal 5g sample.) Refer to Section 17.2 for Michigan project criteria.

8.5.5. Add the correct amount of matrix spiking solution to the matrix spike and matrix spike duplicate samples. (Add 500 µL of 50 µg/mL solution for a nominal 25 g sample, 100 µL for a nominal 5g sample.) Reduce the volume of methanol added to ensure the final volume is 25 mL for nominal 25g sample or 5 mL methanol for a nominal 5g sample. Refer to Section 17.2 for Michigan project criteria.

8.5.6. Prepare an LCS for each batch by adding the correct amount of matrix spiking solution to clean methanol. (500 µL of spike to 25 mL methanol or 100 µL spike to 5 mL methanol). Refer to Section 17.2 for Michigan project criteria.

8.5.7. Shake the samples for two minutes to distribute the methanol throughout the soil.

8.6. Sample collection for medium-level analysis using field methanol preservation

8.6.1. Prepare a 2-oz sample container by adding 25 mL purge and trap grade methanol. (If a 5g sample is to be used, add 5 mL methanol to a 2 oz container or VOA vial).

8.6.2. Seal the bottle and attach a label.

8.6.3. Weigh the bottle to the nearest 0.01g, and note the weight on the label.

8.6.4. Ship with appropriate sampling instructions.

8.6.5. Each sample will require an additional 2 oz plastic bottle with no preservative

for percent moisture determination.

8.6.6. At client request, the methanol addition and weighing may also be performed in the field.

8.6.7. When the samples are returned to the lab, obtain the weight of the soil added to the vial and note on the label.

8.6.8. Add the correct amount of surrogate spiking mixture. (Add 25 µL of 2500 µg/mL solution for a nominal 25g sample, 5 µL for a nominal 5g sample.) Refer to Section 17.2 for Michigan project criteria.

8.6.9. Add the correct amount of matrix spiking solution to the matrix spike and matrix spike duplicate samples. (Add 500 µL of 50 µg/mL solution for a nominal 25g sample, 100 µL for a nominal 5g sample.) Reduce the volume of methanol added to ensure the final volume is 25 mL for nominal 25g sample or 5 mL methanol for a nominal 5g sample. Refer to Section 17.2 for Michigan project criteria.

8.6.10. Prepare an LCS for each batch by adding the correct amount of matrix spiking solution to clean methanol. (500 µL of spike to 25 mL methanol or 100 µL spike to 5 mL methanol). Refer to Section 17.2 for Michigan project criteria.

8.6.11. Shake the samples for two minutes to distribute the methanol throughout the soil.

8.7. Low-level procedure

8.7.1. If low detection limits are required (typically < 50 µg/kg), low-level soil preservation must be used. However, it is also necessary to take a sample for the medium-level (field methanol preserved or using the EnCore™ sampler) procedure in case the concentration of analytes in the soil is above the calibration range of the low-level procedure.

8.7.2. A purge and trap autosampler capable of sampling from a sealed vial is required for analysis of samples collected using this method. (Varian Archon or O.I. 4552).

8.7.3. The soil sample is taken using a 5g EnCore™ sampling device and returned to the lab. It is recommended that two EnCore™ samplers be used for each field sample position to allow for any reruns than may be necessary. A separate sample for % moisture determination is also necessary.

8.7.4. Prepare VOA vials for sodium bisulfate preservation by adding a magnetic stir bar, approximately 1g of sodium bisulfate, and 5 mL of reagent water. Prepare vials for preservation by freezing by adding a stir bar and 5 mL reagent water.

8.7.5. Seal and label the vial. It is strongly recommended that the vial is labeled with an indelible marker rather than a paper label, since paper labels may cause the autosampler to bind and malfunction. The label absolutely must not cover the neck of the vial or the autosampler will malfunction.

8.7.6. Weigh the vial to the nearest 0.1g, and note the weight on the label.

8.7.7. Extrude the soil sample from the EnCore™ sampler into the prepared VOA vial. Reweigh the vial to obtain the weight of soil, and note on the label.

Note: Soils containing carbonates may effervesce when added to the sodium bisulfate solution. If this is the case at a specific site, add 5 mL of water instead, and freeze at <-10°C within 48 hours. The sample must be analyzed within 14 days after sampling and stored at a 45-degree angle in the freezer.

8.7.8. Alternatively the sodium bisulfate preservation may be performed in the field. This is not recommended because of the many problems that can occur in the field setting. Ship at least two vials per sample. The field samplers must determine the weight of soil sampled. Each sample will require an additional 2 oz plastic bottle with no preservative for percent moisture determination, and an additional VOA vial preserved with methanol for the medium level procedure. Depending on the type of soil, it may also be necessary to ship vials with no or extra preservative.

8.8. Unpreserved soils

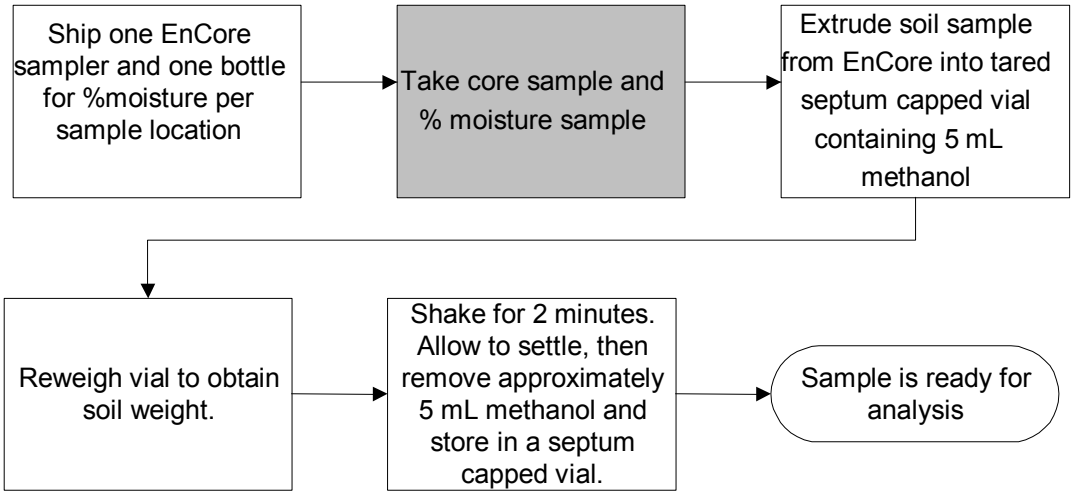
8.8.1. At specific client request, unpreserved soils packed into glass jars or brass tubes may be accepted and sub-sampled in the lab. This is the old procedure based on Method 5030A and Method 8260A. It is no longer included in SW846 and is likely to generate results that are biased low, possibly by more than an order of magnitude.

8.9. Aqueous samples are stored in glass containers with Teflon®-lined septa at 4°C +/- 2°C with minimum headspace.

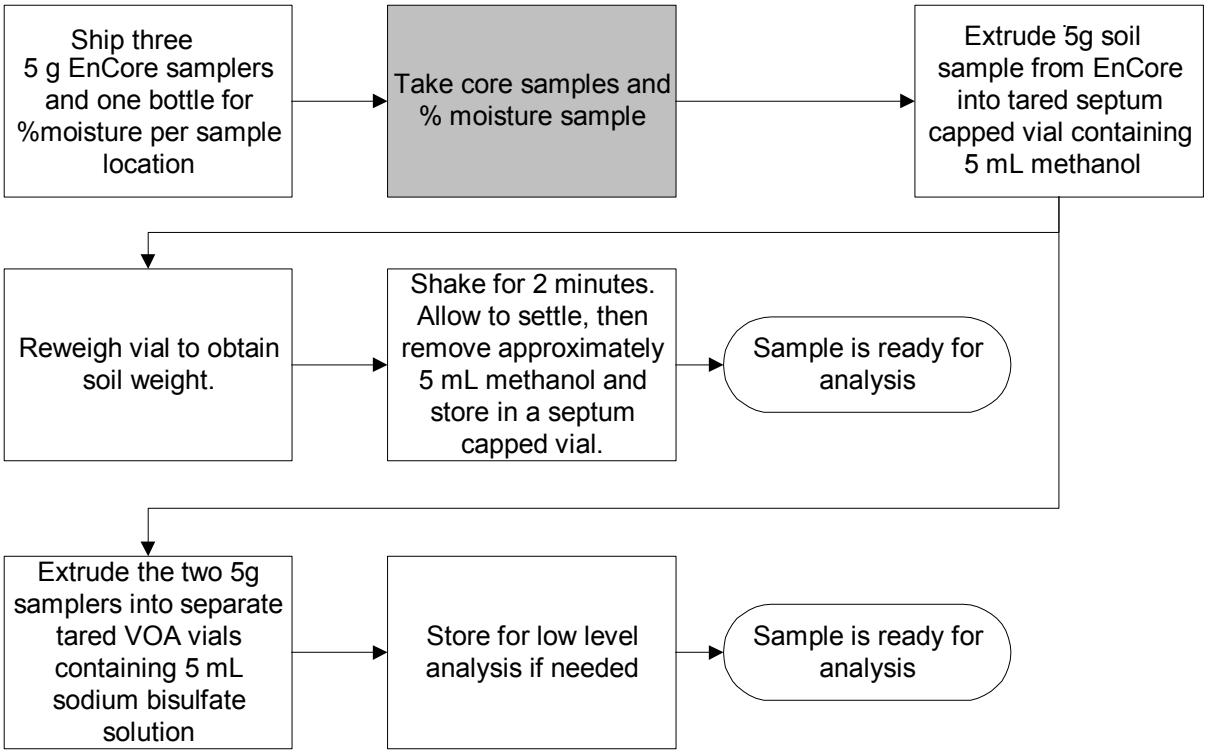
8.10. The maximum holding time is 14 days from sampling until the sample is analyzed. (Samples that are found to be unpreserved still have a 14-day holding time. However, they should be analyzed as soon as possible. The lack of preservation must be addressed in the case narrative). Maximum holding time for the EnCore™ sampler (before the sample is added to methanol or sodium bisulfate) is 48 hours.

8.11. A holding blank is stored with the samples. This is analyzed weekly. It is replaced every seven days.

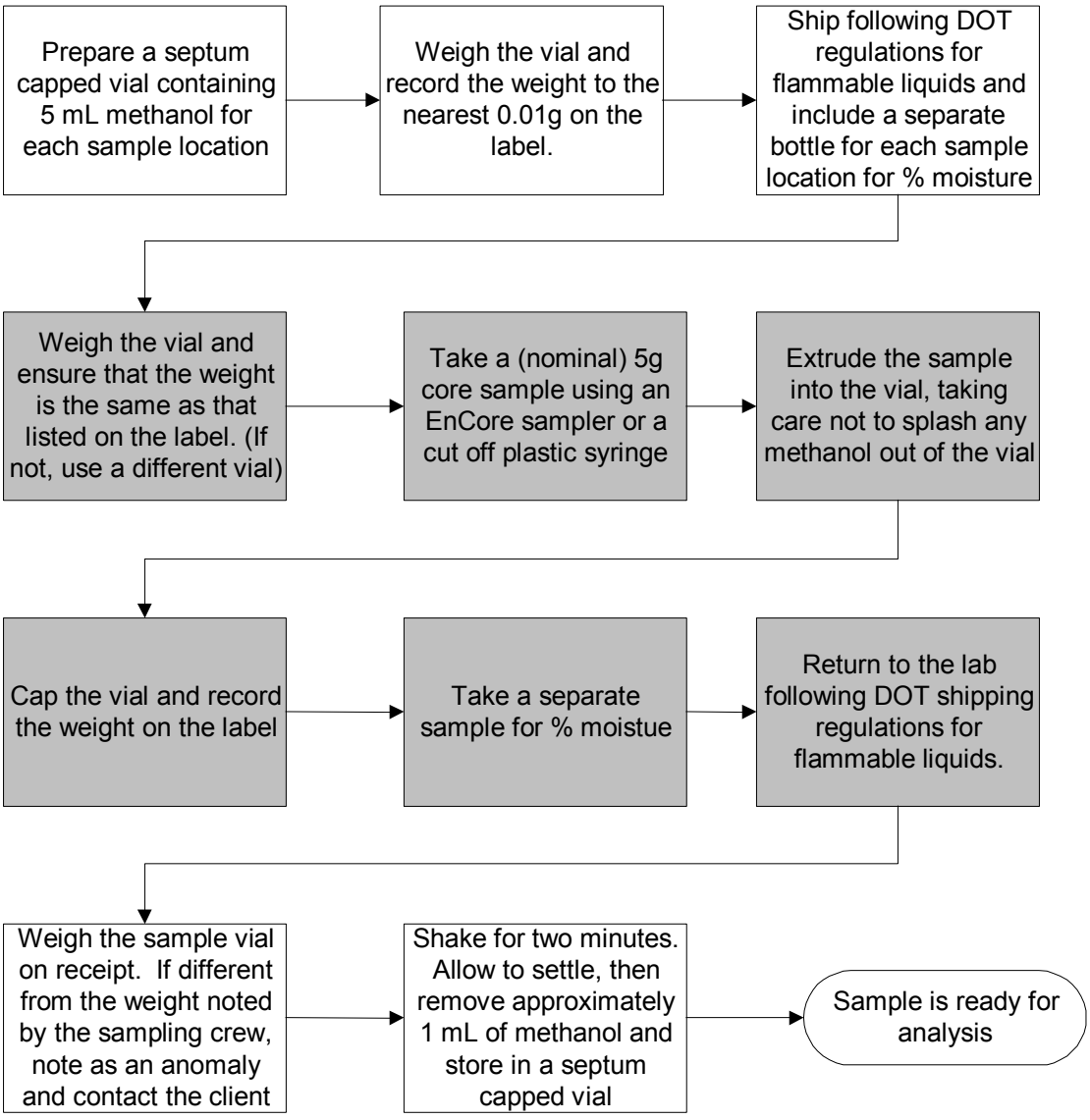
EnCore procedure when low level is not required (field steps in gray)



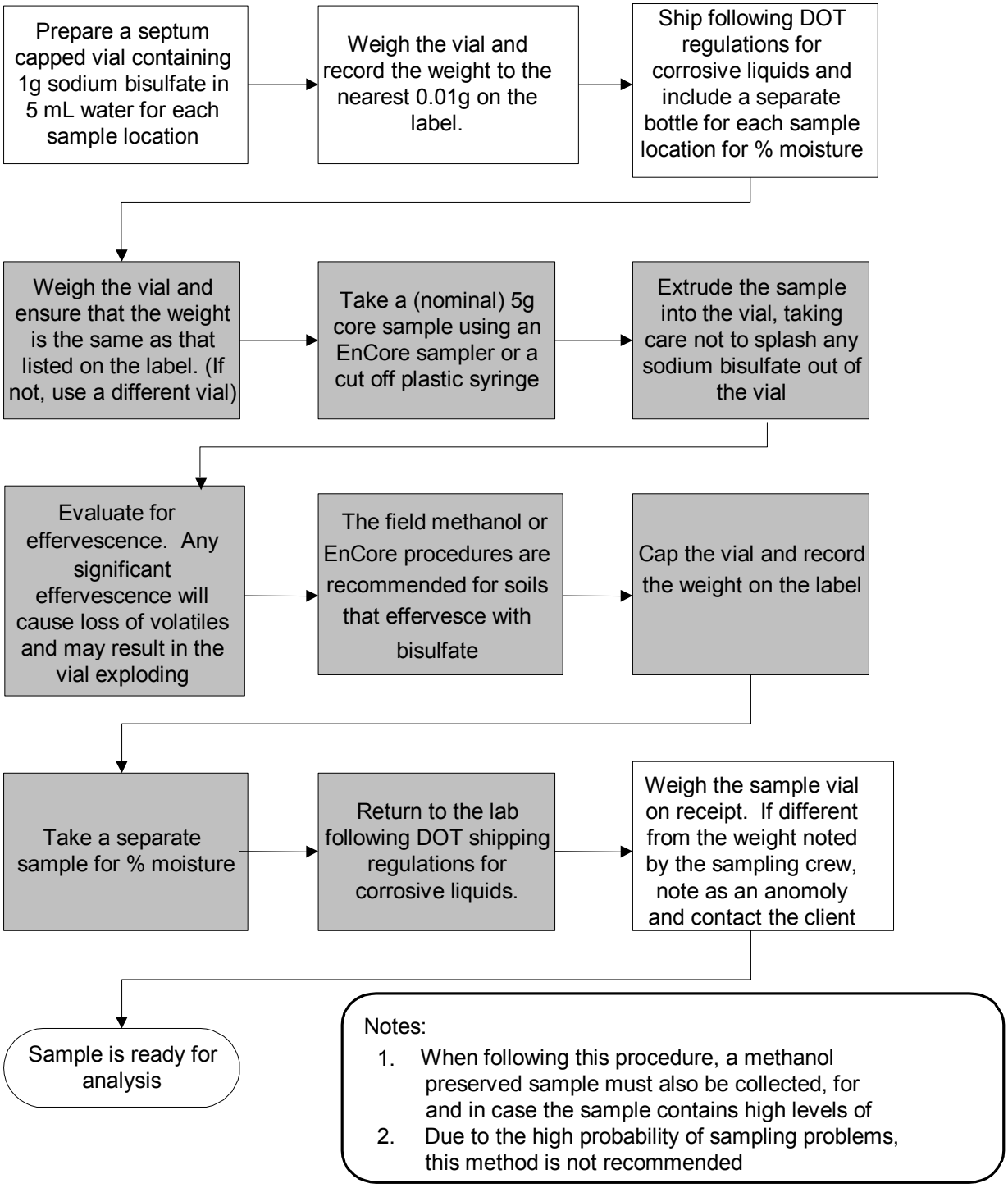
EnCore procedure when low level is required



Field methanol extraction procedure (field steps in gray)



Field bisulfate preservation procedure (field steps in gray)



9. QUALITY CONTROL

9.1. Control Limits

9.1.1. Control limits are established by the laboratory as described in SOP NC-QA-018.

9.1.2. Laboratory control limits are internally generated and updated periodically unless method specified. Control limits are easily accessible via LIMs (QC Browser program).

9.2. Surrogates

9.2.1. Every sample, blank, and QC sample is spiked with surrogates. Surrogate recoveries in samples, blanks, and QC samples must be assessed to ensure that recoveries are within established limits. The compounds included in the surrogate spiking solutions are listed in Table 8. If any surrogates are outside limits, the following corrective actions must take place (except for dilutions):

- Check all calculations for error.
- Ensure instrument performance is acceptable.
- Recalculate the data and/or re-analyze if either of the above checks reveal a problem.
- Reprepare and re-analyze the sample if there is sufficient volume. If there is insufficient volume, the surrogate is narrated.

It is only necessary to reprepare/re-analyze a sample once to demonstrate that poor surrogate recovery is due to matrix effect, unless the analyst believes that the repeated out-of-control results are not due to matrix effect.

9.2.2. If the surrogates are out of control for the sample, matrix spike, and matrix spike duplicate, then matrix effect has been demonstrated for that sample and repreparation is not necessary. If the sample is out of control and the MS and/or MSD is in control, then re-analysis or flagging of the data is required. For Ohio VAP samples, all surrogates must be in control, or samples must be reprepared and re-analyzed.

9.2.3. For concrete matrix, Dibromofluoromethane may have poor recovery in samples and matrix spikes. If the surrogate does not meet criteria, no further action is required due to matrix.

9.2.4. Refer to the TestAmerica QC Program document (QA-003) for further details of

the corrective actions.

9.3. Method Blanks

9.3.1. For each batch of samples, analyze a method blank. The method blank is analyzed after the calibration standards, normally before any samples. For low-level volatiles, the method blank consists of reagent water. For medium-level volatiles, the method blank consists of the same volume of methanol that was used to prepare the samples. Surrogates are added and the method blank is carried through the entire analytical procedure. The method blank must not contain any analyte of interest at or above the reporting limit (except common laboratory contaminants, see below). The method blank is acceptable if any compound detected in the blank is present in the associated samples at ten times the blank level. For Ohio VAP work, there can be no target analyte greater than the RL in the method blank unless the sample result is ND. All samples associated with an unacceptable blank will be reprepared and re-analyzed.

- If the analyte is a common laboratory contaminant (methylene chloride, acetone, 2-butanone), the data may be reported with qualifiers if the concentration of the analyte is less than five times the reporting limit. Such action must be taken in consultation with the client.

- Re-analysis of samples associated with an unacceptable method blank is required when reportable concentrations are determined in the samples.

- If there is no target analyte greater than the RL in the samples associated with an unacceptable method blank, the data may be reported with qualifiers.

9.3.2. The method blank must have acceptable surrogate recoveries. If surrogate recoveries are not acceptable, the data must be evaluated to determine if the method blank has served the purpose of demonstrating that the analysis is free of contamination. If surrogate recoveries are low and there are reportable analytes in the associated samples, re-extraction of the blank and affected samples will normally be required. Consultation with the client must take place. For Ohio VAP samples, all surrogates must be in control, or repreparation of the batch is required.

9.3.3. If reanalysis of the batch is not possible due to limited sample volume or other constraints, the method blank is reported, all associated samples are flagged with a "B," and appropriate comments may be made in a narrative to provide further documentation.

9.3.4. Refer to the TestAmerica QC Program document, QA-003, for further details of the corrective actions.

9.3.5 Refer to SOP NC-QA-016 for further details concerning DoD Project Work.

9.4. Laboratory Control Samples (LCS)

9.4.1. For each batch of samples, analyze an LCS. The LCS is analyzed after the calibration standard, and normally before any samples. The LCS contains a representative subset of the analytes of interest (see Table 9), and must contain the same analytes as the matrix spike. If any analyte or surrogate is outside established control limits, the system is out of control and corrective action must occur. Corrective action will normally be repreparation and re-analysis of the batch. For Ohio VAP samples, all surrogates must be in control on the LCS, or repreparation and re-analysis of the batch is required.

- If the batch is not re-extracted and re-analyzed, the reasons for accepting the batch must be clearly presented in the project records and the report.

- If re-extraction and re-analysis of the batch is not possible due to limited sample volume or other constraints, the LCS is reported, all associated samples are flagged, and appropriate comments are made in a narrative to provide further documentation.

9.4.2. Refer to the TestAmerica QC Program document (QA-003) for further details of the corrective action.

9.4.3. If full analyte spike lists are used at client request, it will be necessary to allow a percentage of the components to be outside control limits as this would be expected statistically. These requirements must be negotiated with the client. n-Hexane must be spiked and reported for the LCS for Ohio VAP samples.

9.4.4. If full analyte spike lists are used at the client request, it is possible some compounds in the LCS may interfere with each other. In that case, the lab will quantitate those compounds in the LCS with a secondary ion which is free from interferences.

9.5. Matrix Spikes

9.5.1. For each QC batch, analyze a matrix spike and matrix spike duplicate. Spiking compounds and levels are given in Table 9. Compare the percent recovery and relative percent difference (RPD) to that in the laboratory-specific, historically-generated limits.

9.5.2. If any individual recovery or RPD falls outside the acceptable range, corrective action must occur. The initial corrective action will be to check the recovery of that analyte in the Laboratory Control Sample (LCS). Generally, if the recovery of the analyte in the LCS is within limits, then the laboratory operation is in control and analysis may proceed. The reasons for accepting the batch must be documented.

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- If the recovery for any component is outside QC limits for both the matrix spike/ spike duplicate and the LCS, the laboratory is out of control and corrective action must be taken. Corrective action will normally include re-analysis of the batch.

- If an MS/MSD is not possible due to limited sample, then an LCS duplicate must be analyzed. RPD of the LCS and LCSD is compared to the matrix spike limits.

- The matrix spike/duplicate must be analyzed at the same dilution as the unspiked sample, even if the matrix spike compounds will be diluted out.

9.6. Non-Conformance and Corrective Action

9.6.1. Any deviations from QC procedures must be documented as a non-conformance with applicable cause and corrective action approved by the facility QA Manager.

10. CALIBRATION AND STANDARDIZATION

10.1. Summary

10.1.1. Prior to the analysis of samples and blanks, each GC/MS system must be tuned and calibrated. Hardware tuning is checked through the analysis of the 4-Bromofluoro-benzene (BFB) to establish that a given GC/MS system meets the standard mass spectral abundance criteria. The GC/MS system must be calibrated initially at a minimum of five concentrations (analyzed under the same BFB tune), to determine the linearity of the response utilizing target calibration standards. Once the system has been calibrated, the calibration must be verified each twelve hour time period for each GC/MS system.

10.1.2. General

Electron Energy:	70 volts (nominal)
Mass Range:	35–300 AMU
Scan Time:	To give at least 5 scans/peak, but not to exceed 2 seconds/scan
Injector Temperature:	200–250°C
Source Temperature:	According to manufacturer's specifications
Transfer Line	Temperature: 250–300°C
Purge Flow:	40 mL/minute
Carrier Gas	Flow: 0.4 – 0.6 mL/minute

10.2 Gas chromatograph suggested temperature program

10.2.1 BFB Analysis

Initial Temperature:	100°C
Initial Hold Time:	0.1 minute
Temperature Program:	20°C/minute
Final Temperature:	200°C

10.2.2 Sample Analysis

Initial Temperature:	40°C
Initial Hold Time:	2minutes
Temperature Program:	15°C/minute
Final Temperature:	200°C
Final Hold Time:	3 minutes

10.3. Instrument Tuning

10.3.1. Each GC/MS system must be hardware-tuned to meet the abundance criteria listed in Table 10 for a maximum of a 50 ng injection or purging of BFB. Analysis must not begin until these criteria are met. These criteria must be met for each 12-hour time period. The 12-hour time period begins at the moment of injection of BFB.

10.4. Initial Calibration

10.4.1. A series of at least five initial calibration standards is prepared and analyzed for the target compounds and each surrogate compound. Six standards must be used for a quadratic least squares calibration. Suggested calibration levels for a 5 mL purge are: 5, 20, 50, 100, and 200 µg/L. Certain analytes are prepared at higher concentrations due to poor purge performance. Suggested calibration levels for a low level 5mL purge are 1, 5, 10, 20, and 40 µg/L. Again, some analytes are prepared at higher levels. Tables 2, 2A, and 4 list the calibration levels for each analyte. Other calibration levels and purge volumes may be used depending on the capabilities of the specific instrument. (For example, adequate sensitivity can be obtained by using a 5 mL purge volume to reach the same reporting limits that once required a 25 mL purge. The calibration levels will still be the same 1, 5, 10, 20, 40 µg/L.) However, the same purge volume must be used for calibration and sample analysis, and the low level standard must be at or below the reporting limit.

10.4.2. It may be necessary to analyze more than one set of calibration standards to encompass all of the analytes required for same tests. For example, the Appendix IX list requires the Primary standard (Table 5) and the Appendix IX standard (Table 6).

10.4.3. Internal standard calibration is used. The internal standards are listed in Table 7. Target compounds must reference the nearest internal standard. Each calibration standard is analyzed and the response factor (RF) for each compound is calculated using the area response of the characteristic ions against the concentration for each

compound and internal standard. See Table 13 for a list of characteristic ions. See Equation 1, Section 12, for calculation of response factor.

10.4.4. The % RSD of the calibration check compounds (CCC) must be less than 30%. Refer to Table 12 for the CCCs. This criteria must be met before sample analysis begins.

10.4.5. The average RF must be calculated for each compound. A system performance check is made prior to using the calibration curve. The five system performance check compounds (SPCC) are checked for a minimum average response factor. Refer to Table 11 for the SPCC compounds and required minimum response factors.

10.4.6. Weighting of data points

10.4.6.1. In a linear or quadratic calibration fit, the points at the lower end of the calibration curve have less weight in determining the curve generated than points at the high concentration end of the curve. However, in environmental analysis, accuracy at the low end of the curve is very important. For this reason, it is preferable to increase the weighting of the lower concentration points. $1/\text{Concentration}^2$ weighting (often called $1/X^2$ weighting) will improve accuracy at the low end of the curve and must be used if the data system has this capability. The Y-intercept is evaluated to determine calibration acceptability.

10.4.7. For any analyte with % RSD >15%, linear or quadratic curve fits may be used if the compounds have historically exhibited a non-linear response. The analyst must consider instrument maintenance to improve the linearity of response. If the % RSD is > 15%, the analyst may drop the low or high in the ICAL , as long as a minimum of five points are maintained (six points for quadratic) and the quantitation range is adjusted accordingly. Otherwise, the coefficient of determination r^2 must be ≥ 0.990.

10.4.8. If time remains in the 12-hour period initiated by the BFB injection before the initial calibration, samples may be analyzed. Otherwise, proceed to continuing calibration.

10.4.9. The calibration standards for the initial five-point calibration for low-level soils that are not preserved in sodium bisulfate (i.e., are preserved by freezing or not preserved) must be heated to 40°C for purging. Using this calibration curve for water samples is acceptable as long as all calibration, QC, and samples are also heated to 40°C. A separate five-point calibration must be prepared for analysis of low level soils that are preserved with sodium bisulfate. Low-level soils analysis requires the use of a closed vial autosampler such as the Varian Archon, O.I. 4552 or Tekmar Precept. Each standard for analysis of sodium bisulfate preserved

samples is prepared by spiking the methanolic standard solution through the septum of a VOA vial containing 5 mL of water and 1g sodium bisulfate. The standards are heated to 40°C for purging. All low-level soil samples, standards, and blanks must also be heated to 40°C for purging.

10.4.10. Non-standard analytes are sometimes requested. For these analytes, it is acceptable to analyze a single standard at the reporting limit with each continuing calibration rather than a five-point initial calibration. If the analyte is detected in any of the samples, a five-point initial calibration must be generated and the sample(s) re-analyzed for quantitation. However, if the analyte is not detected, the non-detect must be reported and no further action is necessary.

Note: This procedure must not be used for Ohio VAP samples.

10.4.11. Calibration accuracy is verified by analyzing a second source standard (ICV) immediately after the initial calibration. The recovery for CCC compounds must be ≤ 20%. The recovery for non-CCC compounds must be ≤ 50% with an allowance of up to six compounds > 50%.

10.5. Continuing Calibration: The initial calibration must be verified every 12 hours.

10.5.1. Continuing calibration begins with analysis of BFB as described in Section 10.3. If the system tune is acceptable, the continuing calibration standard(s) are analyzed. A midpoint calibration standard is used as the continuing calibration.

10.5.2. The RF data from the standards are compared with the average RF from the initial five-point calibration to determine the percent drift of the CCC compounds. The calculation is given in Equation 4, Section 12.3.4.

10.5.3. The % drift of the CCCs must be $\leq$ 20% for the continuing calibration to be valid. The SPCCs are also monitored. The SPCCs must meet the criteria described in Table 11. In addition, the percent drift of all analytes must be $\leq$ 50% with allowance for up to six target analytes to have percent drift > 50%.

10.5.3.1. Refer to Table 12 for specific Ohio VAP analytes.

10.5.4. If the CCCs and/or the SPCCs do not meet the criteria in Section 10.5.3 and Table 11, the system must be evaluated and corrective action must be taken. The BFB tune and continuing calibration must be acceptable before analysis begins. Extensive corrective action such as a different type of column will require a new initial calibration.

10.5.5. Once the above criteria have been met, sample analysis may begin. **Initial calibration average RFs (or the calibration curve) will be used for sample**

quantitation, not the continuing calibration RFs. Analysis may proceed until 12 hours from the injection of the BFB have passed. (A sample *desorbed* less than or equal to 12 hours after the BFB is acceptable.)

11. PROCEDURE

11.1. Procedural Variations

11.1.1. One time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, , chemistry, sample size, or other parameters. Any variation must be completely documented using a Non-Conformance Memo and approved by a Supervisor or Group Leader and QA Manager. The Non-Conformance Memo must be filed in the project file.

11.1.2. Any unauthorized deviations from this procedure must also be documented as a non-conformance with a cause and corrective action described. The laboratory may not deviate from the method for Ohio VAP samples.

11.2. Preliminary Evaluation

11.2.1. Where possible, samples are screened by headspace or GC/MS off-tune analysis to determine the correct aliquot for analysis. Alternatively, an appropriate aliquot can be determined from sample histories.

11.3. Sample Analysis Procedure

11.3.1. All analysis conditions for samples must be the same as for the continuing calibration standards (including purge time and flow, desorb time and temperature, column temperatures, multiplier setting etc.).

11.3.2. All samples must be analyzed as part of a batch. The batch is a set of up to 20 samples of the same matrix processed using the same procedures and reagents within the same time period. The batch also must contain an MS/MSD, an LCS, and a method blank.

11.3.2.1. If there is insufficient time in the 12-hour tune period to analyze 20 samples, the batch may be continued into the next tune period. However, if any re-tuning of the instrument is necessary, or if a period of greater than 24 hours from the preceding BFB tune has passed, a new batch must be started. For medium-level soils, the batch is defined at the sample preparation stage.

11.3.2.2. It is not necessary to re-analyze batch QC with re-analyses of samples. However, any reruns must be part of a valid batch.

11.3.3 Dilutions must be done just prior to the GC/MS analysis of the sample. Dilutions are made in a Luerlok syringe. Calculate the volume of reagent water required for the dilution. Fill the syringe with reagent water, compress the water to vent any residual air and adjust the water volume to the desired amount. Adjust the plunger to the mark and inject the proper aliquot of sample into the syringe. If the dilution required would use less than 1 µL of sample, then serial dilutions must be made in volumetric flasks. Dilutions may also be prepared in a 40 mL vial. An appropriate amount of water is added to the vial. The sample is added using an appropriate syringe.

11.3.3.1 The diluted concentration is to be estimated to be in the upper half of the calibration range.

11.4. Methanol Extract Soils

11.4.1 Rinse a gas-tight syringe with organic free water. Fill the syringe with the same volume of organic free water as used in the calibrations. Add no more than 2% (v/v) (100 µL for a 5 mL purge) methanolic extract (from Sections 8.5 or 8.6) to the syringe. If less than 1µL of methanolic extract is to be added to the water, dilute the methanolic extract such that a volume greater than 1µL will be added to the water in the syringe. Refer to Section 17.2 for Michigan project requirements.

11.5. Liquid wastes that are soluble in methanol and insoluble in water.

11.5.1 Pipette 1 mL of the sample into a tared vial. Use a top-loading balance. Record the weight to the nearest 0.1g.

11.5.2 Quickly add 4 mL of methanol, then add 5µL of a 2500 µg/mL surrogate spiking solution to bring the final volume to 5 mL. Cap the vial and shake for two minutes to mix thoroughly. For an MS/MSD or LCS, 4.9 mL of methanol, 5µL of a 2500 µg/mL surrogate spiking solution, and 0.1 mL of matrix spike solution is used.

11.5.3 Rinse a gas-tight syringe with organic-free water. Fill the syringe with the same volume of organic free water as used in the calibrations. Add no more than 2% (v/v) (100 µL for a 5 mL purge) methanolic extract (from Sections 8.5 or 8.6) to the syringe. If less than 5µL of methanolic extract is to be added to the water, dilute the methanolic extract such that a volume greater than 1µL will be added to the water in the syringe.

11.6. Aqueous and low-level soil sample analysis (Purge and Trap units that sample directly from the VOA vial)

11.6.1 Units which sample from the VOA vial must be equipped with a module which automatically adds surrogate and internal standard solution to the sample prior to

purging the sample.

11.6.2 If the autosampler uses automatic IS/SS injection, no further preparation of the VOA vial is needed. Otherwise, the internal and surrogate standards must be added to the vial. *Note:* Aqueous samples with high amounts of sediment present in the vial may not be suitable for analysis on this instrumentation, or they may need to be analyzed as soils.

11.6.3 Soil samples, which are preserved with sodium bisulfate, must be quantitated against a curve prepared with standards containing about the same amount of sodium bisulfate as the samples (1g in 5 mL).

11.6.4 Soil samples, which are preserved by freezing, must be allowed to thaw completely before sample analysis begins.

11.6.5 Sample remaining in the vial after sampling with one of these mechanisms is no longer valid for further analysis. A fresh VOA vial must be used for further sample analysis.

11.7 Water Samples Not Directly Sampled from VOA Vials

11.7.1. All samples and standard solutions must be at ambient temperature before analysis.

11.7.2. Fill a syringe with the sample. If a dilution is necessary it may be made in the syringe if the sample aliquot is $\geq$ 5 µL. Check and document the pH of the remaining sample.

11.7.3. Add 50 ng of each internal and surrogate standard. The internal standards and the surrogate standards may be mixed and added as one spiking solution (this results in a 10 µg/L solution for a 5 mL sample). Inject the sample into the purging chamber. The internal and surrogate standards can be added automatically by the autosampler.

11.7.3.1. For TCLP samples, use 1 mL of TCLP leachate with 4 mL reagent water. (Note: TCLP reporting limits will be five times higher than the corresponding aqueous limits.)

11.7.4. Purge the sample for 11 minutes (trap must be below 35°C).

11.7.5. After purging is complete, desorb the sample, start the GC temperature program, and begin data acquisition. After desorption, bake the trap for approximately 3-10 minutes to condition it for the next analysis. When the trap is cool, it is ready for the next sample.

11.7.6. Desorb and bake time and temperature are optimized for the type of trap in use. The

same conditions must be used for samples and standards.

11.8. Low-Level Solids Analysis using discrete autosamplers, Methods 8260A and 5030A

Note: This technique may seriously underestimate analyte concentration and must not be used except at specific client request for the purpose of comparability with previous data. It is no longer part of SW-846.

This method is based on purging a heated soil/sediment sample mixed with reagent water containing the surrogates and internal standards. Analyze all reagent blanks and standards under the same conditions as the samples (e.g., heated). The calibration curve is also heated during analysis. Purge temperature is 40°C.

11.8.1. Do not discard any supernatant liquids. Mix the contents of the container with a narrow metal spatula.

11.8.2. Weigh out 5g (or other appropriate aliquot) of sample into a 40 mL vial. Record the weight to the nearest 0.1g. If method sensitivity is demonstrated, a smaller aliquot may be used. Do not use aliquots less than 0.5g. If the sample is contaminated with analytes such that a purge amount less than 0.5g is appropriate, use the medium level method. For the medium level method, add 5g soil to 5 mL methanol containing the surrogates, mix for two minutes, allow to settle, then remove a portion of the methanol, and store in a clean Teflon®-capped vial at 4°C until analysis. Analyze as described in Section 11.5.

11.8.3. Add 5 mL of organic free water to the VOA vial. Add surrogate/internal standard (and matrix spike solutions if required.). Add directly to the sample from Section 11.5.1.

11.8.4. *The above steps must be performed rapidly and without interruption to avoid loss of volatile organics.*

11.9 Medium-Level Soil/Sediment and Waste Samples

11.9.1. Sediments/soils and waste that are insoluble in methanol.

11.9.1.1 Weigh 5 g (wet weight) into a tared vial. Use a top-loading balance. Record the weight to 0.1 gram. Do not discard any supernatant liquids.

11.9.1.2 Quickly add 5 mL of methanol, and 5µL of 2500 µg/mL surrogate spiking solution to bring the final volume of methanol to 5 mL. For an LCS or MS/MSD sample, add 4.9 mL of methanol, 5µL of surrogate spike solution, and 0.1 mL of matrix spike solution. Cap the vial and shake or vortex to mix thoroughly.

Note: Sections 11.9.1.1 and 11.9.1.2 must be performed rapidly and without interruption to avoid the loss of volatile organics.

11.10. Initial review and corrective actions

11.10.1. If the retention time for any internal standard in the continuing calibration changes by more than 0.5 minutes from the mid-level initial calibration standard, the chromatographic system must be inspected for malfunctions and corrected. Re-analysis of samples analyzed while the system was malfunctioning is required.

11.10.2. If the internal standard response in the continuing calibration is more than 200% or less than 50% of the response in the mid-level of the initial calibration standard, the chromatographic system must be inspected for malfunctions and corrected. Re-analysis of samples analyzed while the system was malfunctioning is required. Re-analysis must be undiluted if matrix interference is not observed.

11.10.2.1. Any samples that do not meet the internal standard criteria for the continuing calibration must be evaluated for validity. If the change in sensitivity is a matrix effect, the sample is re-analyzed to confirm. If the change in sensitivity is due to instrumental problems, all affected samples must be re-analyzed after the problem is corrected. For Ohio VAP projects, the laboratory will re-analyze any sample where the internal standard fails, and there is no evidence of matrix interference. If there is no matrix interference, the sample must be reanalyzed at the original dilution. If the internal standard is within criteria, report the second analysis. If the internal standard is still outside of criteria, the sample must be analyzed at a second dilution. If the internal standard still does not meet criteria, the sample must be diluted until the internal standard meets criteria. Multiple runs may be required..

11.11. Dilutions

11.11.1 If the response for any compound exceeds the working range of the GC/MS system, a dilution of the extract is prepared and analyzed. An appropriate dilution must be in the upper half of the calibration range. Samples may be screened to determine the appropriate dilution for the initial run. If the initial diluted run has no hits or hits below 20% of the calibration range and the matrix allows for analysis at a lesser dilution, then the sample must be re-analyzed at a dilution targeted to bring the largest hit above 50% of the calibration range.

11.11.2 Guidance for Dilutions Due to Matrix

11.11.2.1 If the sample is initially run at a dilution and the baseline rise is less

than half the height of the internal standards, or if individual non target peaks are less than twice the height of the internal standards, then the sample must be re-analyzed at a more concentrated dilution. This requirement is approximate and subject to analyst judgement.

11.11.3 Reporting Dilutions

11.11.3.1 The most concentrated dilution with no target compounds above the calibration range will be reported. Other dilutions will only be reported at client request.

12. DATA ANALYSIS AND CALCULATIONS

12.1. Qualitative identification

12.1.1 An analyte is identified by retention time and by comparison of the sample mass spectrum with the mass spectrum of a standard of the suspected compound (standard reference spectrum). Mass spectra for standard reference may be obtained on the user's GC/MS by analysis of the calibration standards or from the NIST Library. Two criteria must be satisfied to verify identification: (1) elution of sample component at the same GC retention time as the standard component, and (2) correspondence of the sample component and the standard component characteristic ions. See Table 13 for a list of the characteristic ions. (Note: Care must be taken to ensure that spectral distortion due to coelution is evaluated.)

- The sample component retention time must compare to within ± 0.2 min. of the retention time of the standard component. For reference, the standard must be run within the same 12 hours as the sample.

- The relative intensities of ions must agree to within ±30% between the standard and sample spectra. (Example: For an ion with an abundance of 50% in the standard spectra, the corresponding sample abundance must be between 20 and 80 percent.)

12.1.2 If a compound cannot be verified by all the above criteria, but in the technical judgment of the analyst, the identification is correct, then the analyst must report that identification and proceed with quantitation.

12.2. Tentatively Identified Compounds (TICs)

12.2.1. If the client requests components not associated with the calibration standards, a search of the NIST library may be made for the purpose of tentative identification. Guidelines are:

12.2.1.1. Relative intensities of major ions in the reference spectrum (ions > 10% of

the most abundant ion) must be present in the sample spectrum.

12.2.1.2. The relative intensities of the major ions must agree to within 20%. (Example: If an ion shows an abundance of 50% in the standard spectrum, the corresponding sample ion abundance must be between 30% and 70%).

12.2.1.3. Molecular ions present in the reference spectrum must be present in the sample spectrum.

12.2.1.4. Ions present in the sample spectrum but not in the reference spectrum must be reviewed for possible background contamination or presence of coeluting compounds.

12.2.1.5. Ions present in the reference spectrum but not in the sample spectrum must be reviewed for possible subtraction from the spectrum because of background contamination or coeluting peaks. (Data system reduction programs can sometimes create these discrepancies.)

12.2.1.6. Computer-generated library search routines must not use normalization routines that would misrepresent the library or unknown spectra when compared to each other. Only after visual inspection of the sample with the nearest library searches must the analyst assign a tentative identification.

12.3. Calculations

12.3.1. Response factor (RF)

Equation 1

$$RF = \frac{A_x C_{is}}{A_{is} C_x}$$

Where:

A_x = Area of the characteristic ion for the compound to be measured

A_{is} = Area of the characteristic ion for the specific internal standard

C_{is} = Concentration of the specific internal standard, ng

C_x = Concentration of the compound being measured, ng

12.3.2. Standard deviation (SD)

Equation 2

$$SD = \sqrt{\sum_{i=1}^{N} \frac{(Xi - X)^2}{N - 1}}$$

Where:

X_i = Value of X at i through N

N = Number of points

X = Average value of X_I

12.3.3. Percent relative standard deviation (%RSD):

Equation 3

$$\%RSD = \frac{\text{Standard Deviation}}{\overline{RF_i}} \times 100$$

$\overline{RF_i}$ = **Mean of RF values in the curve**

12.3.4. Percent drift between the initial calibration and the continuing calibration:

Equation 4

$$\%\ \text{Drift} = \frac{C_{expecte} - C_{found}}{C_{expecte}} \times 100$$

Where:

$C_{expecte}$ = Known concentration in standard

C_{found} = Measured concentration using selected quantitation method

12.3.5. Target compound and surrogate concentrations:

12.3.5.1 Concentrations in the sample may be determined from linear or second order (quadratic) curve fitted to the initial calibration points, or from the average response factor of the initial calibration points. Average response factor may only be used when the % RSD of the response factors in the initial calibration is $\leq$ 15%.

12.3.5.2 Calculation of concentration using Average Response Factors:

Equation 5

$$\text{Concentration } \mu g / L = \frac{x}{RF}$$

12.3.5.3 Calculation of concentration using Linear fit:

Equation 6

$$\text{Concentration } \mu g / L = A + Bx$$

12.3.5.4. Calculation of concentration using Quadratic fit:

Equation 7

$$\text{Concentration } \mu g / L = A + Bx + Cx^2$$

Where,

x is defined in Equations 8, 9, and 10

A is a constant defined by the intercept

B is the slope of the curve

C is the curvature

12.3.5.5. Calculation of x for Water and water-miscible waste:

Equation 8

$$x = \frac{(A_x)(I_s)(D_f)}{(A_{is})(V_o)}$$

Where:

X = ug/L

$\mathbf{A_x}$ = Area of characteristic ion for the compound being measured (secondary ion quantitation is allowed only when there are sample interferences with the primary ion)

$\mathbf{A_{is}}$ = Area of the characteristic ion for the internal standard

$\mathbf{I_s}$ = Amount of internal standard added in ng

$$\text{Dilution Factor} = D_f = \frac{\text{Total volume purged (mL)}}{\text{Volume of original sample used (mL)}}$$

$\mathbf{V_o}$ = Volume of water purged, mL

12.3.5.6. Calculation of ***x*** for Medium level soils:

Equation 9

$$x = \frac{(A_x)(I_s)(V_t)(1000)(D_f)}{(A_{is})(V_a)(W_s)(D)}$$

Where:

X = ug/kg

$\mathbf{A_x}$, $\mathbf{I_s}$, $\mathbf{D_f}$, $\mathbf{A_{is}}$, same as for water.

$\mathbf{V_t}$ = Volume of total extract, mL

$\mathbf{V_a}$ = Volume of extract added for purging, µL

$\mathbf{W_s}$ = Weight of sample extracted, g

$$\mathbf{D} = \frac{100 - \%\text{moisture}}{100}$$

12.3.5.7. Calculation of ***x*** for Low level soils:

Equation 10

$$x = \frac{(A_x)(I_s)}{(A_{is})(W_s)(D)}$$

Where:

X = ug/kg

$\mathbf{A_x}$, $\mathbf{I_s}$, $\mathbf{A_{is}}$, same as for water.

D is as for medium level soils

$\mathbf{W_s}$ = Weight of sample added to the purge vessel, g

12.3.5.8. Calculation of TICs: The calculation of TICs (tentatively identified compounds) is identical to the above calculations with the following exceptions:

$\mathbf{A_x}$ = Area in the total ion chromatogram for the compound being measured

$\mathbf{A_{is}}$ = Area of the total ion chromatogram for the nearest internal standard without interference

RF = 1

In other words, the concentration is equal to ***x*** as defined in Equations 8, 9, and 10.

12.3.6. MS/MSD Recovery

Equation 11

$$\text{Matrix Spike y, \%} = \frac{SSR - SR}{SA} \times 100$$

Where,

SSR = Spike ple result

SR = Sample sult

SA = Spike ed

12.3.7. Relative % Difference calculation for the MS/MSD:

Equation 12

$$\text{RPD} = \frac{|\text{MSR - MSDR}|}{\frac{1}{2}(\text{MSR + MSDR})} \times 100$$

Where:

RPD = Relative percent difference

MSR = Matrix spike result

MSDR = Matrix spike duplicate result

12.4 Additional equations and calculations are listed in the following SOPs: Calibration Curves (General), CA-Q-S-005, and Selection of Calibration Points, CA-T-P-002.

13. METHOD PERFORMANCE

13.1. Method Detection Limit

13.1.1. Generally, each laboratory must generate a valid method detection limit for each analyte of interest. The MDL must be below the reporting limit for each analyte. The procedure for determination of the method detection limit is given in 40 CFR Part 136, Appendix B, and further defined in QA Policy CA-Q-S-006 and SOP NC-QA-021. When non-standard compounds are analyzed at client request, lesser requirements are possible with client agreement. At a minimum, a standard at the reporting limit must be analyzed to demonstrate the capability of the method. The non-standard compound must be detected in the reporting limit standard to be acceptable.

13.1.2. For non-standard analytes, a MDL study must be performed and calibration curve generated before analyzing any samples, unless lesser requirements are previously agreed to with the client. In any event, the minimum initial demonstration required is analysis of a standard at the reporting limit and a single point calibration.

13.2. Initial Demonstration

13.2.1. Each laboratory must have initial demonstration of performance data on file and corresponding method detection limit files.

13.3. Training Qualification

13.3.1. The Group/Team Leader has the responsibility to ensure that this procedure is performed by an analyst who has been properly trained in its use and has the required experience.

13.3.2. Method validation information (where applicable) in the form of laboratory demonstrations of capabilities is maintained for this method in the laboratory QA files.

14. POLLUTION PREVENTION

14.1. It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage, and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention".

15. WASTE MANAGEMENT

15.1. Laboratory personnel assigned to perform hazardous waste disposal procedures must have a working knowledge of the established procedures and practices of TestAmerica. They must have training on the hazardous waste disposal practices upon initial assignment to these tasks, followed by an annual refresher training.

15.2. All waste will be disposed of in accordance with Federal, State, and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention".

15.3. The following waste streams are produced when this method is carried out.

15.3.1. **Acidic material from the auto-sampler**: Waste stream must be collected and

neutralized before discharge to a sewer system if the pH is less than 5.

15.3.2. **Methanol waste from rinses and standards:** Methanol waste is discarded as a flammable liquid in a solvent waste container identified as "Flammable Liquid Waste".

15.3.3. **All samples including purged and extracted soils and waters:** Samples are collected in boxes and removed from the lab to storage. The Waste Coordinator handles crushing the vials and proper disposal.

15.3.4. **Solid samples -** Stirbars are removed from the sample. The contents of the vial are poured into a beaker, and the soil allowed to settle out. The soil is disposed of in the solid waste container.

16. REFERENCES

16.1. References

16.1.1. SW846, *Test Methods for Evaluating Solid Waste,* Third Edition, Gas Chromatography/Mass Spectrometry for Volatile Organics, Method 8260B, Update III, December 1996

16.1.2. SW846, *Test Methods for Evaluating Solid Waste,* Third Edition, Gas Chromatography/Mass Spectrometry for Volatile Organics, Method 8260A, Update II, September 1994

16.1.3. SW846, *Test Methods for Evaluating Solid Waste,* Third Edition, Purge-and-Trap for Aqueous Samples, Method 5030B, Rev 2, December 1996

16.1.4. SW846, *Test Methods for Evaluating Solid Waste,* Third Edition, Purge-and-Trap for Aqueous Samples, Method 5030A, Rev 1, July 1992

16.1.5. SW846, *Test Methods for Evaluating Solid Waste,* Third Edition, Closed System Purge-and-Trap and Extraction for Volatile Organics in Soil and Waste Samples, Method 5035, Rev 0, December 1996

16.1.6 SW846, Test Methods for Evaluating Solid Waste Closed System Purge-and-Trap and Extraction for Volatile Organics in Soil and Waste Samples, Method 5035A, Draft Revision 1, July 2002

16.1.7 TestAmerica North Canton Quality Assurance Manual (QAM), current version

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16.1.8 TestAmerica Corporate Environmental Health and Safety Manual, CW-E-M-001, and TestAmerica North Canton Facility Addendum and Contingency Plan, current version

16.2. Associated SOPs and Policies, latest version

16.2.1. QA Policy, QA-003

16.2.2. Glassware Washing, NC-QA-014

16.2.3. Statistical Evaluation of Data and Development of Control Charts, NC-QA-018

16.2.4. Method Detection Limits and Instrument Detection Limits, CA-Q-S-006 and NC-QA-021

16.2.5. Supplemental Practices for DoD Project Work, NC-QA-016

16.2.6. Standards and Reagents, NC-QA-017

16.2.7. Laboratory Holding Blanks, NC-QA-020

16.2.8. Selection of Calibration Points, CA-T-P-002

16.2.9. Calibration Curves (General), CA-Q-S-005

16.2.10. Acceptable Manual Integration Practices, CA-Q-S-002

16.2.11. Revision History

Historical File:		Revision 2.0: 12/15/97		Revision 2.4: 09/27/04
(formerly CORP-MS-0002NC)		Revision 2.1: 03/06/00		Revision 2.5: 04/03/07
		Revision 2.2: 11/28/00		Revision 0: 06/30/08 (NC-MS-019)
		Revision 2.3: 05/23/01		

17. MISCELLANEOUS

17.1. Modifications from the reference method

17.1.1. A retention time window of 0.2 minutes is used for all components, since some data systems do not have the capability of using the relative retention time units specified in the reference method.

17.1.2. The quantitation and qualifier ions for some compounds have been changed from those recommended in SW846 in order to improve the reliability of qualitative identification.

17.2 The following are protocols that must be followed to achieve the lower reporting limits required when analyzing Michigan projects.

17.2.1 Modify Sections 8.5.4 and 8.6.8 (add 5 uL of 2500 ug/mL surrogate solution for a nominal 10g sample).

17.2.2 Modify Sections 8.5.5 and 8.6.9 (add 100 uL of 50 ug/mL spike solution for a nominal 10g sample).

17.2.3 Modify Sections 8.5.6 and 8.6.10 (add 100 uL of 50 ug/mL spike solution for a nominal 10g sample).

17.2.4 Michigan reporting limits for methanol preserved soils are achieved by injecting 100 uL of the methanol extract in a 5 mL purge. The instrument is calibrated using the recommended calibration range for water of 0.5 ug/L to 100 ug/L. Some analytes are prepared at higher concentrations.

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Table 1 - TestAmerica Primary Standard and Reporting Limits

Compound	CAS Number	Reporting Limits[1]				
		5 mL Water µg/L	Low Level 5 mL water µg/L	Low soil µg/kg	8260B/ 5035 Soil ug/kg	8260A 5030A Med Level Soil µg/kg
Dichlorodifluoromethane	75-71-8	5	1	5	250	250
Chloromethane	74-87-3	5	1	5	250	250
Bromomethane	74-83-9	5	1	5	250	250
Vinyl chloride	75-01-4	5	1	5	250	250
Chloroethane	75-00-3	5	1	5	250	250
Trichlorofluoromethane	75-69-4	5	1	5	250	250
Acrolein	107-02-8	100	20	100	5,000	5,000
Acetone	67-64-1	20	10	20	1,000	1,000
Trichlorotrifluoroethane	76-13-1	5	1	5	250	250
Iodomethane	74-88-4	5	1	5	250	250
Carbon disulfide	75-15-0	5	1	5	250	250
Methylene chloride	75-09-2	5	1	5	250	250
tert-Butyl alcohol	75-65-0	200	50	200	10,000	10,000
1,1-Dichloroethene	75-35-4	5	1	5	250	250
1,1-Dichloroethane	75-34-3	5	1	5	250	250
Trans-1,2-Dichloroethene	156-60-5	5.0	1.0	5.0	250	250
Acrylonitrile	107-13-1	100	20	100	5,000	5,000
Methyl *tert*-butyl ether (MTBE)	1634-04-4	20	5	20	1,000	1,000
Hexane	110-54-3	5	1	5	250	250
cis-1,2-Dichloroethene	156-59-2	5	1	5	250	250
1,2-Dichloroethene (Total)	540-59-0	10	2	10	500	500
Tetrahydrofuran	109-99-9	20	5	20	1,000	1,000
Chloroform	67-66-3	5	1	5	250	250
1,2-Dichloroethane	107-06-2	5	1	5	250	250
Dibromomethane	74-95-3	5	1	5	250	250
2-Butanone	78-93-3	20	5	20	1,000	1,000
1,4-Dioxane	123-91-1	500	200	500	25,000	25,000
1,1,1-Trichloroethane	71-55-6	5	1	5	250	250
Carbon tetrachloride	56-23-5	5	1	5	250	250
Bromodichloromethane	75-27-4	5	1	5	250	250
1,2-Dichloropropane	78-87-5	5	1	5	250	250
cis-1,3-Dichloropropene	10061-01-5	5	1	5	250	250
Trichloroethene	79-01-6	5	1	5	250	250
Dibromochloromethane	124-48-1	5	1	5	250	250
1,2-Dibromoethane	106-93-4	5	1	5	250	250

Table 1 - TestAmerica Primary Standard and Reporting Limits

Compound	CAS Number	Reporting Limits[1]				
		5 mL Water µg/L	Low Level 5 mL water µg/L	Low soil µg/kg	8260B/ 5035 Soil ug/kg	8260A 5030A Med Level Soil µg/kg
1,2,3-Trichloropropane	96-18-4	5	1	5	250	250
1,1,2-Trichloroethane	79-00-5	5	1	5	250	250
Benzene	71-43-2	5	1	5	250	250
Ethylmethacrylate	97-63-2	5	1	5	250	250
Trans-1,3-Dichloropropene	10061-02-6	5	1	5	250	250
Bromoform	75-25-2	5	1	5	250	250
4-Methyl-2-pentanone	108-10-1	20	5	20	1000	1,000
2-Hexanone	591-78-6	20	5	20	1000	1,000
Tetrachloroethene	127-18-4	5	1	5	250	250
Toluene	108-88-3	5	1	5	250	250
1,1,2,2-Tetrachloroethane	79-34-5	5	1	5	250	250
2-Chloroethyl vinyl ether	110-75-8	N/A[2]	N/A	50	1000	1,000
Vinyl acetate	108-05-4	10	2	10	500	500
Chlorobenzene	108-90-7	5	1	5	250	250
Ethylbenzene	100-41-4	5	1	5	250	250
Styrene	100-42-5	5	1	5	250	250
t-1,4-Dichloro-2-butene	110-57-6	5	1	5	250	250
m and p Xylenes		10	2	10	500	500
o-xylene	95-47-6	5.0	1	5	250	250
Total xylenes	1330-20-7	10	2	10	500	500
1,3-Dichlorobenzene	541-73-1	5	1	5	250	250
1,4-Dichlorobenzene	106-46-7	5	1	5	250	250
1,2-Dichlorobenzene	95-50-1	5	1	5	250	250
2,2-Dichloropropane	590-20-7	5	1	5	250	
Bromochloromethane	74-97-5	5	1	5	250	
1,1-Dichloropropene	563-58-6	5	1	5	250	
Bromodichloromethane	75-27-4	5	1	5	250	
1,2-Dichloropropane	78-87-5	5	1	5	250	
1,3-Dichloropropane	142-28-9	5	1	5	250	
Isopropylbenzene	98-82-8	5	1	5	250	
Bromobenzene	108-86-1	5	1	5	250	
n-Propylbenzene	103-65-1	5	1	5	250	
2-Chlorotoluene	95-49-8	5	1	5	250	
4-Chlorotoluene	106-43-4	5	1	5	250	
1,3,5-Trimethylbenzene	108-67-8	5	1	5	250	

Table 1 - TestAmerica Primary Standard and Reporting Limits

Compound	CAS Number	Reporting Limits[1]				
		5 mL Water µg/L	Low Level 5 mL water µg/L	Low soil µg/kg	8260B/ 5035 Soil ug/kg	8260A 5030A Med Level Soil µg/kg
Tert-Butylbenzene	98-06-6	5	1	5	250	
1,2,4-Trimethylbenzene	95-63-6	5	1	5	250	
Sec-butylbenzene	135-98-8	5	1	5	250	
4-Isopropyltoluene	99-87-6	5	1	5	250	
n-Butylbenzene	104-51-8	5	1	5	250	
1,2,4-Trichlorobenzene	120-82-1	5	1	5	250	
Napthalene	91-20-3	5	1	5	250	
Hexachlorobutadiene	87-68-3	5	1	5	250	
1,2,3-Trichlorobenzene	87-61-6	5	1	5	250	
Acetonitrile	75-05-8	100	20	100	5000	
Cyclohexane	110-82-7	10	1	10	500	
Methyl Acetate	79-20-9	10	10	10	500	
Methyl cyclohexane	108-87-2	10	1	10	500	

[1] Reporting limits listed for soil/sediment are based on wet weight. The reporting limits calculated by the laboratory for soil/sediment, calculated on dry weight basis, will be higher.

[2] 2-Chloroethyl vinyl ether cannot be reliably recovered from acid preserved samples

Table 2 - TestAmerica Primary Standard Calibration Levels, 5 mL purge

Compound	Calibration Level ug/L				
	Level 1	Level 2	Level 3	Level 4	Level 5
1,2-Dichloroethane-d4 (Surrogate)	5	20	50	100	200
Toluene-d8 (Surrogate)	5	20	50	100	200
4-Bromofluorobenzene (Surrogate)	5	20	50	100	200
Dichlorodifluoromethane	5	20	50	100	200
Chloromethane	5	20	50	100	200
Bromomethane	5	20	50	100	200
Vinyl chloride	5	20	50	100	200
Chloroethane	5	20	50	100	200
Trichlorofluoromethane	5	20	50	100	200
Acrolein	50	200	500	1000	2000
Acetone	5	20	50	100	200
Trichlorotrifluoroethane	5	20	50	100	200
Iodomethane	5	20	50	100	200
Carbon disulfide	5	20	50	100	200
Methylene chloride	5	20	50	100	200
tert-Butyl alcohol	100	400	1,000	2,000	4,000
1,1-Dichloroethene	5	20	50	100	200
1,1-Dichloroethane	5	20	50	100	200
trans-1,2-Dichloroethene	5	20	50	100	200
Acrylonitrile	50	200	500	1,000	2,000
Methyl *tert*-butyl ether (MTBE)	5	20	50	100	200
Hexane	5	20	50	100	200
cis-1,2-Dichloroethene	5	20	50	100	200
Tetrahydrofuran	5	20	50	100	200
Chloroform	5	20	50	100	200
1,2-Dichloroethane	5	20	50	100	200
Dibromomethane	5	20	50	100	200
2-Butanone	5	20	50	100	200
1,4-Dioxane	250	1000	2,500	5,000	10,000
1,1,1-Trichloroethane	5	20	50	100	200
Carbon tetrachloride	5	20	50	100	200
Bromodichloromethane	5	20	50	100	200
1,2-Dichloropropane	5	20	50	100	200
cis-1,3-Dichloropropene	5	20	50	100	200
Trichloroethene	5	20	50	100	200
Dibromochloromethane	5	20	50	100	200
1,2-Dibromoethane	5	20	50	100	200
1,2,3-Trichloropropane	5	20	50	100	200
Acetonitrile	50	200	500	1000	2000

Table 2 - TestAmerica Primary Standard Calibration Levels, 5 mL purge

Compound	Calibration Level ug/L				
	Level 1	Level 2	Level 3	Level 4	Level 5
1,1,2-Trichloroethane	5	20	50	100	200
Benzene	5	20	50	100	200
Ethylmethacrylate	5	20	50	100	200
trans-1,3-Dichloropropene	5	20	50	100	200
Bromoform	5	20	50	100	200
4-Methyl-2-pentanone	5	20	50	100	200
2-Hexanone	5	20	50	100	200
Tetrachloroethene	5	20	50	100	200
Toluene	5	20	50	100	200
1,1,2,2-Tetrachloroethane	5	20	50	100	200
2-Chloroethyl vinyl ether	10	40	100	200	400
Vinyl acetate	5	20	50	100	200
Chlorobenzene	5	20	50	100	200
Ethylbenzene	5	20	50	100	200
Styrene	5	20	50	100	200
t-1,4-Dichloro-2-butene	5	20	50	100	200
m and p Xylenes	10	40	100	200	400
o-xylene	5	20	50	100	200
1,3-Dichlorobenzene	5	20	50	100	200
1,4-Dichlorobenzene	5	20	50	100	200
1,2-Dichlorobenzene	5	20	50	100	200
2,2-Dichloropropane	5	20	50	100	200
Bromochloromethane	5	20	50	100	200
1,1-Dichloropropene	5	20	50	100	200
Bromodichloromethane	5	20	50	100	200
1,2-Dichloropropane	5	20	50	100	200
1,3-Dichloropropane	5	20	50	100	200
Isopropylbenzene	5	20	50	100	200
Bromobenzene	5	20	50	100	200
n-Propylbenzene	5	20	50	100	200
2-Chlorotoluene	5	20	50	100	200
4-Chlorotoluene	5	20	50	100	200
1,3,5-Trimethylbenzene	5	20	50	100	200
tert-Butylbenzene	5	20	50	100	200
1,2,4-Trimethylbenzene	5	20	50	100	200
sec-butylbenzene	5	20	50	100	200
4-Isopropyltoluene	5	20	50	100	200
n-Butylbenzene	5	20	50	100	200
1,2,4-Trichlorobenzene	5	20	50	100	200
Napthalene	5	20	50	100	200

Table 2 - TestAmerica Primary Standard Calibration Levels, 5 mL purge

Compound	Calibration Level ug/L				
	Level 1	Level 2	Level 3	Level 4	Level 5
Hexachlorobutadiene	5	20	50	100	200
1,2,3-Trichlorobenzene	5	20	50	100	200

Table 2A - TestAmerica Primary Standard Calibration Levels, Low Level [1]

Compound	Calibration Level ug/L				
	Level 1	Level 2	Level 3	Level 4	Level 5
Dibromofluoromethane (Surrogate)	1	5	10	20	40
1,2-Dichloroethane-d4 (Surrogate)	1	5	10	20	40
Toluene-d8 (Surrogate)	1	5	10	20	40
Bromofluorobenzene (Surrogate)	1	5	10	20	40
Dichlorodifluoromethane	1	5	10	20	40
Chloromethane	1	5	10	20	40
Vinyl Chloride	1	5	10	20	40
Bromomethane	1	5	10	20	40
Chloroethane	1	5	10	20	40
Trichlorofluoromethane	1	5	10	20	40
Acrolein	10	50	100	200	400
Acetone	2	10	20	40	80
1,1-Dichloroethene	1	5	10	20	40
Trichlorotrifluoroethane	1	5	10	20	40
Iodomethane	1	5	10	20	40
Carbon Disulfide	1	5	10	20	40
Methylene Chloride	1	5	10	20	40
Acetonitrile	10	50	100	200	400
Acrylonitrile	10	50	100	200	400
Methyl tert-butyl ether	1	5	10	20	40
trans-1,2-Dichloroethene	1	5	10	20	40
Hexane	1	5	10	20	40
Vinyl acetate	1	5	10	20	40
1,1-Dichloroethane	1	5	10	20	40
tert-Butyl Alcohol	20	100	200	400	800
2-Butanone	2	10	20	40	80
cis-1,2-dichloroethene	1	5	10	20	40
2,2-Dichloropropane	1	5	10	20	40
Bromochloromethane	1	5	10	20	40
Chloroform	1	5	10	20	40

Table 2A - TestAmerica Primary Standard Calibration Levels, Low Level [1]

Compound	Calibration Level ug/L				
	Level 1	Level 2	Level 3	Level 4	Level 5
Tetrahvdrofuran	1	5	10	20	40
1,1,1-Trichloroethane	1	5	10	20	40
1,1-Dichloropropene	1	5	10	20	40
Carbon Tetrachloride	1	5	10	20	40
1,2-Dichloroethane	1	5	10	20	40
Benzene	1	5	10	20	40
Trichloroethene	1	5	10	20	40
1,2-Dichloropropane	1	5	10	20	40
1,4-Dioxane	50	250	500	1000	2000
Dibromomethane	1	5	10	20	40
Bromodichloromethane	1	5	10	20	40
2-Chloroethyl vinyl ether	2	10	20	40	80
cis-1,3-Dichloropropene	1	5	10	20	40
4-Methyl-2-pentanone	2	10	20	40	80
Toluene	1	5	10	20	40
trans-1,3-Dichloropropene	1	5	10	20	40
Ethyl Methacrylate	1	5	10	20	40
1,1,2-Trichloroethane	1	5	10	20	40
1,3-Dichloropropane	1	5	10	20	40
Tetrachloroethene	1	5	10	20	40
2-Hexanone	2	10	20	40	80
Dibromochloromethane	1	5	10	20	40
1,2-Dibromoethane	1	5	10	20	40
Chlorobenzene	1	5	10	20	40
1,1,1,2-Tetrachloroethane	1	5	10	20	40
Ethylbenzene	1	5	10	20	40
m + p-Xylene	2	10	20	40	80
Xylene-o	1	5	10	20	40
Styrene	1	5	10	20	40
Bromoform	1	5	10	20	40
Isopropylbenzene	1	5	10	20	40
1,1,2,2-Tetrachloroethane	1	5	10	20	40
1,4-Dichloro-2-butene	1	5	10	20	40
1,2,3-Trichloropropane	1	5	10	20	40
Bromobenzene	1	5	10	20	40
n-Propylbenzene	1	5	10	20	40
2-Chlorotoluene	1	5	10	20	40
1,3,5-Trimethylbenzene	1	5	10	20	40
4-Chlorotoluene	1	5	10	20	40

Table 2A - TestAmerica Primary Standard Calibration Levels, Low Level [1]

Compound	Calibration Level ug/L				
	Level 1	Level 2	Level 3	Level 4	Level 5
tert-Butylbenzene	1	5	10	20	40
1,2,4-Trimethylbenzene	1	5	10	20	40
sec-Butylbenzene	1	5	10	20	40
4-Isopropyltoluene	1	5	10	20	40
1,3-Dichlorobenzene	1	5	10	20	40
1,4-Dichlorobenzene	1	5	10	20	40
n-Butylbenzene	1	5	10	20	40
1,2-Dichlorobenzene	1	5	10	20	40
1,2-Dibromo-3-chloropropane	1	5	10	20	40
1,2,4-Trichlorobenzene	1	5	10	20	40
Hexachlorobutadiene	1	5	10	20	40
Naphthalene	1	5	10	20	40
1,2,3-Trichlorobenzene	1	5	10	20	40
Cyclohexane	1	5	10	20	40
Methyl Acetate	2	10	20	40	80
Methylcyclohexane	1	5	10	20	40
1,3,5-Trichlorobenzene	1	5	10	20	40

[1] 25 mL purge samples analyzed at 5 mL purge on more sensitive equipment.

Table 3 - TestAmerica Appendix IX Standard and Reporting Limits, 5 mL purge

Compound	CAS Number	Reporting Limits			
		5 mL Water µg/L	Low Level 5mL purge water µg/L	Low Soil µg/kg	Medium Soil µg/mL
Allyl Chloride	107-05-1	10	2	10	500
Dichlorofluoromethane	75-43-4	10	2	10	500
Isopropyl ether	108-20-3	10	2	10	500
Chloroprene	126-99-8	5	2	5	250
n-Butanol	71-36-3	200	50	200	10,000
Propionitrile	107-12-0	20	4	20	1000
Methacrylonitrile	126-98-7	5	2	5	250
Isobutanol	78-83-1	200	50	200	10,000
Methyl methacrylate	80-62-6	5	2	5	250
1,1,1,2-Tetrachloroethane	630-20-6	5	1	5	250
1,2-Dibromo-3-chloropropane	96-12-8	10	2	10	500
Ethyl ether	60-29-7	10	2	10	500
Ethyl Acetate	141-78-6	20	4	20	1,000
2-Nitropropane	79-46-9	10	4	10	500
Cyclohexanone	108-94-1	50	20	50	2500
Isopropylbenzene	98-82-8	5	1	5	250
2-Methylnaphthalene (Michigan only)	91-57-6	NA	5	NA	330

Table 4

Recommended/TestAmerica Appendix IX Standard Calibration Levels, µg/L

Compound	Level 1	Level 2	Level 3	Level 4	Level 5
Allyl Chloride	5	20	50	100	200
Dichlorofluoromethane	5	20	50	100	200
Isopropyl ether	5	20	50	100	200
Chloroprene	5	20	50	100	200
n-Butanol	100	400	1,000	2,000	4,000
Propionitrile	10	40	100	200	400
Methacrylonitrile	5	20	50	100	200
Isobutanol	100	400	1,000	2,000	4,000
Methyl methacrylate	5	20	50	100	200
1,1,1,2-Tetrachloroethane	5	20	50	100	200
1,2-Dibromo-3-chloropropane	10	40	100	200	400
Ethyl ether	5	20	50	100	200
Ethyl Acetate	10	40	100	200	400
2-Nitropropane	10	40	100	200	400
Cyclohexanone	50	200	500	1,000	2,000
2-Methylnaphthalene (Michigan only)	2	10	20	40	80
Ethyl tert-butyl ether	5	20	50	100	200
tert-Amyl methyl ether	5	20	50	100	200
1,2,3-Trimethylbenzene	5	20	50	100	200

Table 5 - Reportable Analytes for TestAmerica Standard Tests, Primary Standard

Compound	CAS Number	TestAmerica Standard List	TCLP	TCL	Appendix IX
Dichlorodifluoromethane	75-71-8			X	X
Chloromethane	74-87-3	X		X	X
Bromomethane	74-83-9	X		X	X
Vinyl chloride	75-01-4	X	X	X	X
Chloroethane	75-00-3	X		X	X
Trichlorofluoromethane	75-69-4			X	X
Acrolein	107-02-8				X
Acetone	67-64-1	X		X	X
Trichlorotrifluoroethane	76-13-1				
Iodomethane	74-88-4				X
Carbon disulfide	75-15-0	X		X	X
Methylene chloride	75-09-2	X		X	X
tert-Butyl alcohol	75-65-0				
1,1-Dichloroethene	75-35-4	X	X	X	X
1,1-Dichloroethane	75-34-3	X		X	X
trans-1,2-Dichloroethene	156-60-5	X		X	X
Total 1,2-Dichloroethene		X		X	X
Acrylonitrile	107-13-1				X
Methyl *tert*-butyl ether (MTBE)	1634-04-4			X	
Hexane	110-54-3				
cis-1,2-Dichloroethene	156-59-2	X		X	
Tetrahydrofuran	109-99-9				
Chloroform	67-66-3	X	X	X	X
1,2-Dichloroethane	107-06-2	X	X	X	X
Dibromomethane	74-95-3				X
2-Butanone	78-93-3	X	X	X	X
1,4-Dioxane	123-91-1				X
1,1,1-Trichloroethane	71-55-6	X		X	X
Carbon tetrachloride	56-23-5	X	X	X	X
Bromodichloromethane	75-27-4	X		X	X
1,2-Dichloropropane	78-87-5	X		X	X
cis-1,3-Dichloropropene	10061-01-5	X		X	X
Trichloroethene	79-01-6	X	X	X	X
Dibromochloromethane	124-48-1	X		X	X
1,2-Dibromoethane	106-93-4			X	X
1,2,3-Trichloropropane	96-18-4				X
1,1,2-Trichloroethane	79-00-5	X		X	X

Table 5 - Reportable Analytes for TestAmerica Standard Tests, Primary Standard

Compound	CAS Number	TestAmerica Standard List	TCLP	TCL	Appendix IX
Benzene	71-43-2	X	X	X	X
Ethylmethacrylate	97-63-2				X
trans-1,3-Dichloropropene	10061-02-6	X		X	X
Bromoform	75-25-2	X		X	X
4-Methyl-2-pentanone	108-10-1	X		X	X
2-Hexanone	591-78-6	X		X	X
Tetrachloroethene	127-18-4	X	X	X	X
Toluene	108-88-3	X		X	X
1,1,2,2-Tetrachloroethane	79-34-5	X		X	X
2-Chloroethyl vinyl ether	110-75-8				
Vinyl acetate	108-05-4				X
Chlorobenzene	108-90-7	X	X	X	X
Ethylbenzene	100-41-4	X		X	X
Styrene	100-42-5	X		X	X
t-1,4-Dichloro-2-butene	110-57-6				X
m and p Xylenes		X		X	X
o-xylene	95-47-6	X		X	X
Total xylenes	1330-20-7	X		X	X
1,3-Dichlorobenzene	541-73-1	X		X	
1,4-Dichlorobenzene	106-46-7	X		X	
1,2-Dichlorobenzene	95-50-1	X		X	
Cyclohexane	110-82-7	X		X	
Methyl Acetate	79-20-9	X		X	
Methyl cyclohexane	108-87-2	X		X	
Isopropylbenzene	98-82-8			X	
1,2-Dibromo-3-chloropropane	96-12-8			X	X
1,2,4-Trichlorobenzene	120-82-1			X	
Acetonitrile	75-05-8				X
1,1,1,2-Tetrachloroethane	630-20-6				X
2,2-Dichloropropene	594-20-7				
Bromochloromethane	74-97-5				
1,1-Dichloropropene	563-58-6				
1,3-Dichloropropane	142-28-9				
Bromobenzene	108-86-1				
n-Propylbenzene	103-65-1				
2-Chlorotoluene	95-49-8				
1,3,5-Trimethylbenzene	108-67-8				
4-Chlorotoluene	106-43-4				
tert-Butylbenzene	98-06-6				
1,2,4-Trimethylbenzene	95-63-6				

Table 5 - Reportable Analytes for TestAmerica Standard Tests, Primary Standard

Compound	CAS Number	TestAmerica Standard List	TCLP	TCL	Appendix IX
sec-Butylbenzene	135-98-8				
4-Isopropyltoluene	99-87-6				
n-Butylbenzene	104-51-8				
Hexachlorobutadiene	87-68-3				
Naphthalene	91-20-3				
1,2,3-Trichlorobenzene	87-61-6				
1,3,5-Trichlorobenzene	108-70-3				

Table 6

Reportable Analytes for TestAmerica Standard Tests, Appendix IX standard

Compound	Number	Appendix IX
Allyl Chloride	107-05-1	X
Dichlorofluoromethane	75-43-4	
Isopropyl ether	108-20-3	
Chloroprene	126-99-8	X
n-Butanol	71-36-3	
Propionitrile	107-12-0	X
Methacrylonitrile	126-98-7	X
Isobutanol	78-83-1	X
Methyl methacrylate	80-62-6	X
Ethyl ether	60-29-7	
Ethyl Acetate	141-78-6	
2-Nitropropane	79-46-9	
Cyclohexanone	108-94-1	
Ethyl tert-butyl ether	637-92-3	
tert-Amyl methyl ether	994-05-8	
1,2,3-trimethylbenzene	526-73-8	

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Table 7 - Internal Standards

Compound	Standard Concentration µg/mL (may vary per matrix)	Quantitation ion (5 mL purge)
Fluorobenzene	50 – 250	96
Chlorobenzene-d5	50 – 250	117
1,4-Dichlorobenzene-d4	50 – 250	152

Notes:

1) Except for medium level soils, the surrogate and internal standards may be combined in one solution.

Table 8 - Surrogate Standards

Surrogate Compounds	Standard Concentration µg/mL (may vary per matrix)
1,2-Dichloroethane-d_4	50 – 250
Dibromofluoromethane	50 – 250
Toluene-d_8	50 - 250
4-Bromofluorobenzene	50 – 250

Notes:

1) Except for medium level soils, the surrogate and internal standards may be combined in one solution.

2) Recovery limits for surrogates are generated from historical data and are maintained by the QA Dept.

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Table 9 - Matrix Spike / LCS Control Compounds

Compound	Standard Concentration µg /mL
1,1,1-Trichloroethane	50 - 250
1,1,2,2-Tetrachloroethane	50
1,1,2-Trichloro-1,2,2-trifluoroethane	50
1,1,2-Trichloroethane	50
1,1-Dichloroethane	50
1,1-Dichloroethene	50
1,1-Dichloropropene	50
1,2,3-Trichlorobenzene	50
1,2,3-Trichloropropane	50
1,2,4-Trichlorobenzene	50
1,2,4-Trimethylbenzene	50
1,2-Dibromo-3-chloropropane	50
1,2-Dibromoethane	50
1,2-Dichlorobenzene	50
1,2-Dichloroethane	50
1,2-Dichloroethene (total)	100
1,2-Dichloropropane	50
1,3,5-Trimethylbenzene	50
1,3-Dichlorobenzene	50
1,3-Dichloropropane	50
1,4-Dichlorobenzene	50
2,2-Dichloropropane	50
2-Butanone	50
2-Chloroethyl Vinyl Ether	100 - 500
2-Chlorotoluene	50
2-Hexanone	50
4-Chlorotoluene	50
4-Methyl-2-pentanone	50
Acetone	50
Acetonitrile	500 – 2500
Acrolein	500
Acrylonitrile	100 - 500
Benzene	50
Bromobenzene	50
Bromochloromethane	50
Bromodichloromethane	50
Bromoform	50
Bromomethane	50
Carbon disulfide	50
Carbon tetrachloride	50
Chlorobenzene	50
Chloroethane	50

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Table 9 - Matrix Spike / LCS Control Compounds

Compound	Standard Concentration µg /mL
Chloroform	50
Chloromethane	50
cis-1,2-Dichloroethene	50
cis-1,3-Dichloropropene	50
Cyclohexane	50
Dibromochloromethane	50
Dibromomethane	50
Dichlorodifluoromethane	50
Ethylbenzene	50
Hexachlorobutadiene	50
Iodomethane	50
Isopropylbenzene	50
Isopropylether	50
Methyl acetate	50
Methyl tert-butyl ether (MTBE)	50
Methylcyclohexane	50
Methylene chloride	50
Naphthalene	50
n-Butylbenzene	50
n-Hexane (Ohio VAP only)	50
n-Propylbenzene	50
p-Isopropyltoluene	50
sec-Butylbenzene	50
Styrene	50
tert-Butylbenzene	50
Tetrachloroethene	50
Toluene	50
trans-1,2-Dichloroethene	50
trans-1,2-Dichloroethene	50
trans-1,3-Dichloropropene	50
Trichloroethene	50
Trichlorofluoromethane	50
Vinyl Acetate	50
Vinyl chloride	50
Xylenes (total)	150 - 750

Notes: 1) 5 µL of the standard is added to the LCS or matrix spiked sample. This results in a concentration of each spike analyte in the sample of 50µg/L for a 5 mL purge or 10 µg/L for a 25 mL purge.

2) Recovery and precision limits for LCS and MS/MSD are generated from historical data and are maintained by QA Dept.

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Table 10 - BFB Key Ion Abundance Criteria

Mass	Ion Abundance Criteria
50	15% to 40% of Mass 95
75	30% to 60% of Mass 95
95	Base Peak, 100% Relative Abundance
96	5% to 9% of Mass 95
173	Less Than 2% of Mass 174
174	Greater Than 50% of Mass 95
175	5% to 9% of Mass 174
176	Greater Than 95%, But Less Than 101% of Mass 174
177	5% to 9% of Mass 176

Table 11 - SPCC Compounds and Minimum Response Factors

Compound	8260B, 8260A Min. RF
Chloromethane	0.100
1,1-Dichloroethane	0.100
Bromoform	0.100
1,1,2,2-Tetrachloroethane	0.300
Chlorobenzene	0.300

Table 12 - CCC compounds

Compound	Max. %RSD from Initial Calibration	Max. %D for continuing calibration
Vinyl Chloride	30	20
1,1-Dichloroethene	30	20
Chloroform	30	20
1,2-Dichloropropane	30	20
Toluene	30	20
Ethylbenzene	30	20
n-Hexane (Ohio VAP only)	30	20

Table 13 - Characteristic Ions

Compound	Primary*	Secondary	Tertiary
1,2-Dichloroethane-d_4 (Surrogate)	65	102	
Dichlorodifluoromethane	85	87	50, 101,103
Chloromethane	50	52	49
Vinyl chloride	62	64	61
Bromomethane	94	96	79
Chloroethane	64	66	49
Trichlorofluoromethane	101	103	66
1,1-Dichloroethene	96	61	98
Acrolein	56	55	58
Iodomethane	142	127	141
Carbon disulfide	76	78	
Trichlorotrifluoroethane	151	101	153
Acetone	43	58	
Methylene chloride	84	49	51, 86
tert-Butyl alcohol	59	74	
trans-1,2-Dichloroethene	96	61	98
Acrylonitrile	53	52	51
Methyl *tert* butyl ether	73		
Hexane	57	43	
1,1-Dichloroethane	63	65	83
cis-1,2-Dichloroethene	96	61	98
2-Butanone	43	72**	
Tetrahydrofuran	42	71	
Chloroform	83	85	47
1,2-Dichloroethane	62	64	98

Table 13 - Characteristic Ions

Compound	Primary*	Secondary	Tertiary
Dibromomethane	93	174	95, 172, 176
1,4-Dioxane	88	58	
Vinyl acetate	43	86	
1,1,1-Trichloroethane	97	99	117
Carbon tetrachloride	117	119	121
Benzene	78	52	77
Trichloroethene	130	95	97, 132
1,2-Dichloropropane	63	65	41
Bromodichloromethane	83	85	129
2-Chloroethyl vinyl ether	63	65	106
cis-1,3-Dichloropropene	75	77	39
trans-1,3-Dichloropropene	75	77	39
1,1,2-Trichloroethane	97	83	85, 99
Chlorodibromomethane	129	127	131
Bromoform	173	171	175, 252
1,2,3-Trichloropropane	75	110	77, 112, 97
Toluene-d_8 (Surrogate)	98	70	100
4-Bromofluorobenzene (Surrogate)	95	174	176
Toluene	91	92	65
4-Methyl-2-pentanone	43	58	57, 100
Tetrachloroethene	164	166	131
Ethyl methacrylate	69	41	99, 86, 114
2-Hexanone	43	58	57, 100
Chlorobenzene	112	114	77
Ethylbenzene	106	91	
Xylenes	106	91	
Styrene	104	103	78, 51, 77
Dichlorobenzene (all isomers)	146	148	111
trans 1,4-Dichloro-2-butene	53	75	89, 77, 124
1,1,2,2-Tetrachloroethane	83	85	131, 133
Allyl Chloride	76	41	78
Acetonitrile	40	41	
Dichlorofluoromethane	67	69	
Isopropyl ether	87	59	45
Chloroprene	53	88	90
n-Butanol	56	41	42
Propionitrile	54	52	55
Methacrylonitrile	41	67	52
Isobutanol	41	43	74
Methyl methacrylate	41	69	100

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Table 13 - Characteristic Ions

Compound	Primary*	Secondary	Tertiary
1,1,1,2-Tetrachloroethane	131	133	119
1,2-Dibromo-3-chloropropane	157	155	75
Ethyl ether	59	74	
Ethyl Acetate	43	88	61
2-Nitropropane	41	43	46
Cyclohexanone	55	42	98
Isopropylbenzene	105	120	
Cyclohexane	56	69	84
Methyl Acetate	43	74	
Methyl cyclohexane	83	55	98

* The primary ion must be used for quantitation unless interferences are present, in which case a secondary ion may be used.

** m/z 43 may be used for quantitation of 2-Butanone, but m/z 72 must be present for positive identification.

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Title: GC/MS ANALYSIS BASED ON METHODS 8270C AND 8270D

[Method: SW846 8270C and 8270D]

Approvals (Signature/Date):

Technology Specialist	10/07/10 Date	Health & Safety Coordinator	10/11/10 Date
Quality Assurance Manager	10/20/10 Date	Technical Director	10/07/10 Date
Laboratory Director	10/07/10 Date		

This SOP was previously identified as SOP NC-MS-018, Rev 1, dated 12/16/08

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1. SCOPE AND APPLICATION

1.1 This method is based upon SW846 8270C and 8270D, and is applicable to the determination of the concentration of semivolatile organic compounds in extracts prepared from solid and aqueous matrices. Direct injection of a sample may be used in limited applications. Refer to Tables 1 and 2 for the list of compounds applicable for this method. Note that the compounds are listed in approximate retention time order. Additional compounds may be amenable to this method. If non-standard analytes are required, they must be validated by the procedures described in Section 13 before sample analysis.

1.2 The following compounds may require special treatment when being determined by this method:

- Benzidine can be subject to oxidative losses during solvent concentration and exhibits poor chromatography. Neutral extraction should be performed if this compound is expected.
- Hexachlorocyclopentadiene is subject to thermal decomposition in the inlet of the gas chromatograph, chemical reaction in acetone solution, and photochemical decomposition.
- N-Nitrosodiphenylamine decomposes in the gas chromatographic inlet and cannot be distinguished from diphenylamine.
- Pentachlorophenol, 2,4-dinitrophenol, 4-nitrophenol, 4,6-dinitro-2-methylphenol, 4-chloro-3-methylphenol, benzoic acid, 2-nitroaniline, 3-nitroaniline, 4-chloroaniline, and benzyl alcohol are subject to erratic chromatographic behavior, especially if the GC system is contaminated with high boiling material.
- Hexachlorophene is not amenable to analysis by this method.
- 3-Methylphenol cannot be separated from 4-methylphenol by the conditions specified in this method.

1.3 The standard reporting limit of this method for determining an individual compound is approximately 0.33 mg/kg (wet weight) for soil/sediment samples, 1 - 200 mg/kg for wastes (dependent on matrix and method of preparation), and 10 µg/L for groundwater samples. Some compounds have higher reporting limits. Refer to Tables 1 and 2 for specific reporting limits. Reporting limits will be proportionately higher for sample extracts that require dilution.

2. SUMMARY OF METHOD

2.1 Aqueous samples are extracted with methylene chloride using a separatory funnel and/or a continuous extractor. Solid samples are extracted with methylene chloride / acetone using sonication, or soxhlet. The extract is dried, concentrated to a final volume of 2 mL for waters and soils, and analyzed by GC/MS. Extraction procedures are detailed in SOP NC-OP-032

2.2 The semivolatile compounds are introduced into the GC/MS by injecting the sample extracted into a gas chromatograph equipped with a narrow-bore fused silica capillary column. The GC column is temperature-programmed to separate the analytes, which are then detected with a mass spectrometer (MS) connected to the gas chromatograph.

2.3 Analytes eluted from the capillary column are introduced into the mass spectrometer via a jet separator or a direct connection. Identification of target analytes is accomplished by comparing their mass spectra with the electron impact spectra of authentic standards. Quantitation is accomplished by comparing the response of a major (quantitation_ ion relative to an internal standard using an appropriate calibration curve for the intended application.

3. DEFINITIONS

3.1 Refer to the TestAmerica North Canton Quality Assurance Manual (QAM), current version, for definitions of terms used in this document.

4. INTERFERENCES

4.1 Method interferences may be caused by contaminants in solvents, reagents, glassware, and other processing apparatus that lead to discrete artifacts. All of these materials must be routinely demonstrated to be free from interferences under conditions of the analysis by running laboratory method blanks as described in the Quality Control section. Raw GC/MS data from all blanks, samples, and spikes must be evaluated for interferences. If an interference is detected, it is necessary to determine if the source of interference is in the preparation and/or cleanup of the samples; then take corrective action to eliminate the problem.

4.2 The use of high purity reagents, solvents, and gases helps to minimize interference problems.

4.3 Matrix interferences may be caused by contaminants that are co-extracted from the sample. The extent of matrix interferences will vary considerably from source to source, depending upon the nature of the sample.

4.4 Contamination by carryover can occur whenever high-level and low-level samples are sequentially analyzed. To reduce carryover, the sample syringe must be rinsed with solvent between samples. Whenever an unusually concentrated sample is encountered, it must be followed by the analysis of solvent to check for cross contamination.

4.5 Phthalate contamination is commonly observed in this analysis and its occurrence must be carefully evaluated as an indicator of a contamination problem in the sample preparation step of the analysis.

5. SAFETY PRECAUTIONS

5.1 Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual, the Facility Addendum to the Corporate EH&S Manual, and this document.

5.2 Eye protection that protects against splash, laboratory coat, and appropriate gloves must be worn while samples, standards, solvents and reagents are being handled. Disposable gloves that have become contaminated must be removed and discarded; other gloves must be cleaned immediately.

5.3 Chemicals that have been classified as carcinogens, or potential carcinogens, under OSHA

include Benzo(a)anthracene, benzidine, 3,3'-dichlorobenzidine, benzo(a)pyrene, dibenzo(a,h)anthracene, and n-nitrosodimethylamine.

5.4 The following is a list of the materials used in this method, which have a serious or significant hazard rating. **NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the Reagents and Standards section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Methylene Chloride	Carcinogen Irritant	25 ppm-TWA 125 ppm-STEL	Causes irritation to respiratory tract. Has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting and headache. Causes irritation, redness and pain to the skin and eyes. Prolonged contact can cause burns. Liquid degreases the skin. May be absorbed through skin.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

5.5 It is recommended that analysts break up work tasks to avoid repetitive motion tasks, such as opening a large number of vials or containers in one time period.

5.6 Exposure to chemicals must be maintained as low as reasonably achievable. All samples with stickers that read “Caution/Use Hood!” must be opened in the hood. Contact the EH&S Coordinator if this is not possible. Solvent and waste containers must be kept closed unless transfers are being made.

5.7 The preparation of standards and reagents must be conducted in a fume hood with the sash closed as far as the operation will permit.

5.8 It is recommended that neat standards be purchased only as a last resort. The preparation of standards from neat materials and reagents must be conducted in a fume hood with the sash closed as far as the operations will permit.

5.9 Standards in solution may be diluted in the open laboratory when syringes and the like are utilized.

5.10 All work must be stopped in the event of a known or potential compromise to the health and safety of a TestAmerica North Canton associate. The situation must be reported immediately to a Laboratory Supervisor and the EH&S Coordinator.

6. EQUIPMENT AND SUPPLIES

6.1 Gas Chromatograph/Mass Spectrometer System: An analytical system complete with a

temperature-programmable gas chromatograph suitable for split/splitless injection and all required accessories, including syringes, analytical columns, and gases. The capillary column must be directly coupled to the source.

6.2 Column: 20m x 0.18mm ID, 0.36µm film thickness silicon-coated fused-silica capillary column (J & W Scientific DB-5.625 or equivalent). Alternate columns are acceptable if they provide acceptable performance.

6.3 Mass Spectrometer: Capable of scanning from 35 to 500 AMU every one second or less, using 70 volts (nominal) electron energy in the electron impact ionization mode. The mass spectrometer must be capable of producing a mass spectrum for decafluorotriphenylphosphine (DFTPP) which meets all of the criteria in Table 4 when the GC/MS tuning standard is injected through the GC.

6.4 GC/MS Interface: Any GC-to-MS interface that gives acceptable calibration points and achieves acceptable tuning performance criteria may be used.

6.5 Data System: A computer system must be interfaced to the mass spectrometer. The system must allow the continuous acquisition and storage on machine-readable media of all mass spectra obtained throughout the duration of the chromatographic program. The computer must have software that can search any GC/MS data file for ions of a specific mass and that can plot such ion abundances versus time or scan number. This type of plot is defined as the Extracted Ion Current Profile (EICP). Software must also be available that allows integrating the abundances in any EICP between specified time or scan-number limits. The most recent version of the EPA/NIH Mass Spectral Library is recommended.

6.6 Syringe: 5 µL Hamilton Laboratory grade syringes or equivalent.

6.7 Carrier gas: Ultra high purity helium.

6.8 Autosampler vials, inserts, and caps

7. REAGENTS AND STANDARDS

7.1 A minimum five-point calibration curve is prepared. The standard preparation information is detailed in the Standard Logbook. If a quadratic regression is used, six points must be analyzed for the calibration curve. The low point must be at or below the reporting limit. Refer to Table 10 for typical calibration levels for all analytes. Other calibration levels may be used, depending on instrument capability, but the low standard must support the reporting limit and the high standard defines the range of the calibration. For Ohio VAP work, the low standard must be at, or below, the reporting limit.

7.2 An Internal Standard solution is prepared by diluting a purchased standard. The standard preparation information is detailed in the Standard Logbook. Compounds in the I.S. Mix are acenaphthene-d10, chrysene-d12, 1,4-dichlorobenzene-d4, naphthalene-d8, perylene-d12, and phenanthrene-d10.

7.3 Surrogate Standard Spiking Solution: Prepare as indicated in the preparative methods. Preparation information is detailed in the Standard Logbook for the Organic Prep group. See appropriate preparation SOP. Surrogate compounds and levels are listed in Table 9.

7.4 GC/MS Tuning Standard: A methylene chloride solution containing decafluorotriphenylphosphine (DFTPP) is prepared. The standard preparation information is detailed in the Standard Logbook. Pentachlorophenol, benzidine, and DDT, must also be included in the Tuning Standard. All components are at 25 ug/mL.

7.5 The standards listed in Sections 7.1 to 7.4 must be refrigerated at $\leq 6^{o}C$ when not in use. Refrigeration at $-10^{o}C$ to $-20^{o}C$ may be used if it can be demonstrated that analytes do not fall out of solution at this temperature. The standards must be replaced at least once a year. Additional information can be found in SOP NC-QA-017.

8. SAMPLE PRESERVATION AND STORAGE

8.1 Sample extracts are stored at $4 \pm 2^{o}C$. Samples and extracts must be stored in suitable glass containers with Teflon®-lined caps. (Extracts will be stored for 30 days after invoicing.)

8.2 Water samples are extracted within seven days of sampling, and the extracts are analyzed within 40 days of extraction. Solids, sludges, and organic liquids are extracted within 14 days of sampling and the extracts are analyzed within 40 days of extraction.

9. QUALITY CONTROL

9.1 Initial Demonstration of Capability

9.1.1 For the standard analyte list, the initial demonstration and method detection limit (MDL) studies described in Section 13 must be acceptable before analysis of samples may begin.

9.1.2 For non-standard analytes, an MDL study must be performed and calibration curve generated before analyzing any samples, unless lesser requirements are previously agreed to with the client. In any event, the minimum initial demonstration required is analysis of an extracted standard at the reporting limit and a single point calibration. For DoD projects, the initial demonstration must include an MDL study analysis of the LCS replicates and a minimum of five-point calibration.

9.2 Control Limits

9.2.1 In-house historical control limits must be determined for surrogates, matrix spikes, and laboratory control samples (LCS). These limits must be determined periodically. Control limits are established by the laboratory as described in SOP NC-QA-018. Control limits are easily accessible via LIMs (QC Browser program).

9.2.2 If samples are diluted, the surrogate and matrix spike recoveries must be reported with a DIL flag. For DoD projects, all surrogates must be within control limits.

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9.2.3 All surrogate, LCS, and MS recoveries (except for dilutions) must be entered into LIMS (when available) or other database so that accurate historical control limits can be generated.

9.2.4 Refer to the QC Program document (QA-003) for further details of control limits.

9.3 Batch - The batch is a set of up to 20 samples of the same matrix processed using the same procedures and reagents within the same time period. The Quality Control batch must contain a matrix spike / spike duplicate (MS/MSD), a Laboratory Control Sample (LCS), and a method blank. Batches are defined at the sample preparation stage. Batches must be kept together through the whole analytical process to the extent possible, but it is not mandatory to analyze prepared extracts on the same instrument or in the same sequence. Refer to the TestAmerica North Canton QC Program document (QA-003) for further details of the batch definition.

9.4 Method Blank

9.4.1 A method blank is prepared and analyzed with each batch of samples. The method blank consists of reagent water for aqueous samples and sodium sulfate for soil samples (refer to SOP NC-OP-032 for details). Surrogates are added and the method blank is carried through the entire analytical procedure. The method blank must not contain any analyte of interest at or above the reporting limit (except common lab contaminants, see below). Any blank contamination above the reporting limit must be less than 1/10 of the measured concentration of any sample in the associated preparation batch. Refer to SOP NC-QA-016 for DoD requirements.

9.4.1.1 If the analyte is a common laboratory contaminant (phthalate esters), the data may be reported with qualifiers if the concentration of the analyte is less than five times the RL. Such action must be taken in consultation with the client.

9.4.1.2 Re-analysis of any samples with reportable concentrations of analytes found in the method blank is required unless other actions are agreed with the client.

9.4.1.3 If there is no target analyte greater than the RL in the samples associated with an unacceptable method blank, the data may be reported with qualifiers. Such action should be taken in consultation with the client. NOTE: For Ohio VAP work, there can be no target analyte greater than the RL in the method blank. All samples associated with an unacceptable blank must be re-extracted and re-analyzed. The exceptions are as follows: (a) insufficient sample for re-extraction/redigestion, (b) expired holding times, or (c) the analytes detected in the Method Blank are non-detect in the associated samples.

9.4.2 The method blank must have acceptable surrogate recoveries. If surrogate recoveries are not acceptable, the data must be evaluated to determine if the method blank has served the purpose of demonstrating that the analysis is free of contamination. If surrogate recoveries are low and there are reportable analytes in the associated samples, re-extraction of the blank and affected samples will normally be required. Consultation with the client must

take place. For Ohio VAP samples, all analytes must meet criteria or the samples must be re-extracted if sufficient volume of sample remains.

9.4.3 If re-analysis of the batch is not possible due to limited sample volume or other constraints, the method blank is reported, all associated samples are flagged with a "B", and appropriate comments must be made in a narrative to provide further documentation.

9.4.4 Refer to the TestAmerica North Canton QC Program document (QA-003) for further details of the corrective actions.

9.5 Laboratory Control Sample (LCS)

9.5.1 A laboratory control sample (LCS) is prepared and analyzed with every batch of samples. All control analytes must be within established control limits. The LCS is spiked with the compounds listed in Table6 unless specified by a client or agency.

9.5.2 If any control analyte in the LCS is outside the laboratory established historical control limits, corrective action must occur. All non-controlling compounds must attain a recovery of 10% or greater if the compound is on the client's list. Corrective action may include re-extraction and re-analysis of the batch. For Ohio VAP samples, all analytes must meet criteria or the samples must be re-extracted if sufficient volume of sample remains.

9.5.2.1 If the batch is not re-extracted and re-analyzed, the reasons for accepting the batch must be clearly presented in the project records and the report. (An example of acceptable reasons for not re-analyzing might be that the matrix spike and matrix spike duplicate are acceptable, and sample surrogate recoveries are good, demonstrating that the problem was confined to the LCS).

9.5.2.2 If re-extraction and re-analysis of the batch is not possible due to limited sample volume or other constraints, the LCS is reported, all associated samples are flagged, and appropriate comments are made in a narrative to provide further documentation.

9.5.2.3 The LCS must have acceptable surrogate recoveries. If surrogate recoveries are low, re-extraction of the LCS and affected samples will normally be required. Consultation with the client should take place. For Ohio VAP samples, all analytes must meet criteria or the samples must be re-extracted. The exceptions are as follows: (a) insufficient sample for re-extraction/re-digestion, (b) expired holding times, or (c) the LCS is biased high and the samples are non-detect for those analytes.

9.5.3 Ongoing monitoring of the LCS over time provides evidence that the laboratory is performing the method within accepted QC guidelines for accuracy and precision.

9.6 Matrix Spike/Matrix Spike Duplicate (MS/MSD)

9.6.1 A matrix spike/matrix spike duplicate (MS/MSD) is prepared and analyzed with every

batch of samples. The MS/MSD is spiked with the same subset of analytes as the LCS (See Table6). Compare the percent recovery and relative percent difference (RPD) to that in the laboratory specific historically-generated limits.

9.6.1.1 If the recovery for any component is outside QC limits for both the matrix spike / spike duplicate and the LCS, the laboratory is out of control and corrective action must be taken. Corrective action must include re-preparation and re-analysis of the batch.

9.6.1.2 If an MS/MSD is not possible due to limited sample, then an LCS duplicate must be analyzed. RPD of the LCS and LCSD are compared to in-house limits.

9.6.1.3 The matrix spike / duplicate must be analyzed at the same dilution as the unspiked sample, even if the matrix spike compounds will be diluted out.

9.7 Surrogates

9.7.1 Every sample, blank, and QC sample is spiked with surrogate standards. Surrogate spike recoveries must be evaluated by determining whether the concentration (measured as percent recovery) falls within the required recovery limits. The compounds routinely included in the surrogate spiking solution, along with recommended standard concentrations, are listed in Table 9.

9.7.2 If any surrogates are outside limits, the following corrective actions must take place (except for dilutions):

9.7.2.1 Check all calculations for error.

9.7.2.2 Ensure that instrument performance is acceptable.

9.7.2.3 Recalculate the data and/or re-analyze the extract if either of the above checks reveal a problem.

9.7.2.3.1 It is only necessary to reprepare / re-analyze a sample once to demonstrate that poor surrogate recovery is due to matrix effect, unless the analyst believes that the repeated out of control results are not due to matrix effect.

Note: If all associated QC meets criteria (blank, LCS/LCSD), up to one surrogate per fraction may be outside of acceptance criteria , as long as the recovery is greater than 10%. **Note:** For Ohio VAP and DoD samples, all surrogates must be within acceptance criteria. The exceptions for Ohio VAP are as follows: (a) insufficient sample for re-extraction, or (b) the surrogates are biased high and the samples are non-detect.

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9.7.3 If the sample with surrogate recoveries outside the recovery limits was a sample used for an MS/MSD and the surrogate recoveries in the MS/MSD are also outside of the control limits, then the sample, the MS, and the MSD do not require re-analysis as this phenomenon would indicate a possible matrix problem.

9.7.4 If the sample is re-analyzed and the surrogate recoveries in the re-analysis are acceptable, then the problem was within the analyst's control and only the re-analyzed data must be reported (unless the re-analysis was outside holding times, in which case, reporting both sets of results may be appropriate).

9.7.5 If the re-analysis does confirm the original results, the original analysis is reported and the data flagged as estimated due to matrix effect.

9.8 Nonconformance and Corrective Action

9.8.1 Any deviations from QC procedures must be documented as a nonconformance with applicable cause and corrective action approved by the facility QA Manager.

10. CALIBRATION AND STANDARDIZATION

10.1 Summary

10.1.1 The instrument is tuned for DFTPP, calibrated initially with a minimum five-point calibration curve, and verified each 12-hour shift with one or more continuing calibration standard(s). Recommended instrument conditions are listed in Table 3.

10.1.2 For DoD work, refer to SOP NC-QA-016 for specific details.

10.2 All standards and extracts are allowed to warm to room temperature before injecting.

10.3 Instrument Tuning

10.3.1 At the beginning of every 12-hour shift when analyses are to be performed, the GC/MS system must be checked to see if acceptable performance criteria (Table 4) is achieved for DFTPP (decafluorotriphenylphosphine).

10.3.2 Inject the GC/MS tuning standard (Section 7.4) into the GC/MS system. Obtain a background-corrected mass spectra of DFTPP and confirm that all the key m/z criteria in Table 4 are achieved. If all the criteria are not achieved, the analyst must retune the mass spectrometer and repeat the test until all criteria are achieved. The performance criteria must be achieved before any samples, blanks, or standards are analyzed.

10.3.3 The GC/MS tuning standard must also be used to evaluate the inertness of the chromatographic system. The tailing factor for benzidine must be less than 3.0. The tailing factor for pentachlorophenol must be less than 5. For Method 8270D, benzidine and pentachlorochlorophenol should be present at their normal responses, and should not

exceed a tailing factor of 2. DDT must be included in the tuning standard, and its breakdown must be < 20%. Refer to Section 12 for the appropriate calculations.

10.4 Initial Calibration

10.4.1 Internal Standard Calibration Procedure. Internal standards are listed in Table 5. Use the base peak m/z as the primary m/z for quantitation of the standards. If interferences are noted, use one of the next two most intense masses for quantitation.

10.4.2 Compounds should be assigned to the IS with the closest retention time. Refer to Table 11 for internal standard corresponding analytes.

10.4.3 Prepare calibration standards at a minimum of five concentration levels for each parameter of interest. Six standards must be used for a quadratic least squares calibration. It may also be useful to analyze six calibration levels and use the lower five for most analytes and the upper five for analytes that have poor response. Add the internal standard mixture to result in 2 ng on column. (For example, 5 uL of 80ppm IS mix is added to 100 uL of extract. This results in 4 ng; but only 0.5 uL is injected, resulting in a final on column amount of 2 ng.). The concentration ranges of all analytes are listed in Table 10. For Ohio VAP work, the low standard must be at or below the reporting limit.

10.4.4 Analyze each calibration standard and tabulate the area of the primary characteristic m/z against concentration for each compound and internal standard. Table 5 lists the analytes and characteristic ions analyzed in the laboratory. Calculate response factors (RF), average response factors, and the percent RSD of the response factors for each compound using the equations in Section 12. For Method 8270C, verify that the SPCC and CCC criteria in Sections 10.4.5 and 10.4.7 are met. **No sample analysis must be performed unless these criteria are met.**

10.4.5 System Performance Check Compounds (SPCCs) (Method 8270C). The minimum average RF for semivolatile SPCCs is 0.050. If the minimum response factors are not met, the system must be evaluated and corrective action must be taken before sample analysis begins. Some possible problems are standard mixture degradation, injection port inlet contamination, contamination at the front end of the analytical column, and active sites in the column or chromatographic system. This check must be met before analysis begins.

SPCC Compounds:

N-nitroso-di-n-propylamine
Hexachlorocyclopentadiene
2,4-Dinitrophenol
4-Nitrophenol

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10.4.6 Initial Calibration Criteria for Method 8270D

10.4.6.1 The RSD should be less than 20% for each analyte. For analytes that fail, use linear or quadratic curve with 0.99 correlation coefficient.

NOTE: If compliance with Method 8270C is required, the RSD limit is 15%.

10.4.6.2 No more than 10% of compounds can fail the 20%/0.99 correlation requirement.

10.4.6.3 If more than 10% of analytes fail both 20% RSD and 0.99 correlation, then recalibration is necessary.

10.4.6.4 Any individual analyte that fails both 20% RSD and 0.99 correlation must have any positive result flagged as estimated (or can be noted in the narrative).

10.4.6.5 For any analyte non-detect associated with a calibration that fails the 20% RSD/0.99 correlation/minimum response factor criteria, there must be a demonstration of adequate sensitivity at the quantitation limit.

10.4.6.6 Minimum response factor should be met, especially for the low level standard.

10.4.6.7 Any individual analyte that fails the minimum response factor set in the SOP must have a demonstration of sensitivity in the analytical batch to report non-detects. The demonstration of sensitivity is analysis of a low level CCV (at or below the reporting limit). The criterion for a passing LLCCV is detection only, and a passing LLCCV allows non-detects to be reported without flagging. The low level CCV would normally be analyzed immediately after the mid-level CCV. In general, Table 4 in the method should be used as guidance in setting minimum response factors in the SOP; but the RFs may be modified if appropriate (for example, if especially low-level analysis is performed).

10.4.6.8 For Method 8270D, the minimum response factors are listed in Table 12.

10.4.7 Calibration Check Compounds (CCCs) (Method 8270C). The %RSD of the response factors for each CCC in the initial calibration must be less than 30% for the initial calibration to be considered valid. This criterion must be met before sample analysis begins. Problems similar to those listed under SPCCs could affect this criterion.

10.4.7.1 If none of the CCCs are required analytes, project-specific calibration specifications must be agreed with the client.

10.4.7.2 CCC Compounds

Phenol

Acenaphthene
1,4-Dichlorobenzene
N-nitrosodiphenylamine
2-Nitrophenol
Pentachlorophenol
2,4-Dichlorophenol
Fluoranthene
Hexachlorobutadiene
Di-n-octylphthalate
4-Chloro-3-methylphenol
Benzo(a)pyrene
2,4,6-Trichlorophenol

10.4.7.3 Continuing Calibration Criteria for Method 8270D

10.4.7.3.1 At least 80% of analytes must have a %D less than or equal to 20%.

10.4.7.3.2 Minimum response factors must be evaluated.

10.4.8 If the software in use is capable of routinely reporting curve coefficients for data validation purposes, and the necessary calibration reports can be generated, then the analyst must evaluate analytes with %RSD > 15% for calibration on a curve. If it appears that substantially better accuracy would be obtained using quantitation from a curve, then the appropriate curve with no forced intercept must be used for quantitation.

10.4.8.1 If an analyte in the initial calibration is > 15%, then calibration on a curve must be used. Quadratic curve fits must be used if the compound has historically exhibited a nonlinear response. The analyst must consider instrument maintenance to improve the linearity of response. Use of $1/Concentration^2$ weighting is recommended to improve the accuracy of quantitation at the low end of the curve. If Relative Standard Error (RSE) is used to evaluate the curve, it must be better than 15%. If the % RSD is >15%, the analyst may drop the low or high points in the ICAL, as long as a minimum of five points are maintained and the quantitation range is adjusted accordingly. If the % RSD is still >15%, a quadratic or linear curve must be used. The coefficient of determination (r^2) must be ≥ 0.990. If the coefficient of determination is < 0.990, then any hits for these compounds must be flagged as estimated. If a curve is not linear for any compound that is found in a sample, the result must be flagged as estimated. Linear is defined as <15% RSD or a coefficient of determination of 0.990.

Note: For Method 8270D, analytes using the linear calibration fit should have the read back concentration of the low level standard evaluated. The read back concentration should be within 30% of the true value. Any sample detects for analytes that fail the read back criterion and are using a linear calibration must be flagged as estimated, or described in the narrative.

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Note: For Ohio VAP work, the low standard must be at or below the reporting limit.

Note: Several components do not respond well by this method (poor linearity). These compounds are indene, acrylamide, 4-Nitroquinoline-1-oxide, famphur, benzenethiol, kepone, and 2,4-toluenediamine. If these compounds are requested by a client and hits are found, alternate standards or methods will be needed for more accurate quantitation. Sensitivity as demonstrated by the low standard is sufficient to substantiate a non-detect.

10.4.8.2 If time remains in the 12-hour period initiated by the DFTPP injection before the initial calibration, samples must be analyzed. Otherwise, proceed to continuing calibration.

10.4.8.3 Quantitation is performed using the calibration curve or average response factor from the initial curve.

10.5 Initial Calibration Verification (ICV)

10.5.1 Calibration accuracy is verified by analyzing a second source standard (ICV) immediately after the initial calibration. The recovery CCC compounds must be $\leq$ 20%. The recovery for non-CCC compounds must be $\leq$ 50% with an allowance of up to six compounds >50%.

10.5.2 For Method 8270D, the suggested acceptance limit is 70-130% for all analytes.

10.6 Continuing Calibration

10.6.1 At the start of each 12-hour period, analyze a GC/MS tuning standard. The injection of DFTPP must result in a mass spectrum for DFTPP which meets the criteria given in Table 4.

10.6.2 Following a successful DFTPP analysis the continuing calibration standard(s) are analyzed. The standards must contain all semivolatile analytes, including all required surrogates. A mid-level calibration standard is used for the continuing calibration.

10.6.3 For Method 8270C, the following criteria must be met for the continuing calibration to be acceptable:

- The SPCC compounds must have a response factor of $\geq$ 0.05.
- The percent difference or drift of the CCC compounds from the initial calibration must be $\leq$ 20% (see Section 12 for calculations). In addition, the percent difference or drift of all analytes must be $\leq$ 50%, with allowance for up to four compounds to be greater than 50%.

- The internal standard response must be within 50-200% of the response in the mid level of the initial calibration.

- The internal standard retention times must be within 30 seconds of the retention times in the mid-level of the initial calibration.

  Note: There is no internal standard criteria for samples. Criteria is only for continuing and initial calibrations.

  Note: Ohio VAP requires that any sample with internal standard outliers be re-analyzed undiluted unless a matrix effect is observed. If there is no matrix interference, the sample must be re-analyzed at the original dilution. If the internal standard is within criteria, report the second analysis. If the internal standard is still outside of criteria, the sample must be diluted until the internal standard meets criteria. Multiple runs may be required. The criteria for acceptance is between 50% and 200% of the same internal standard in continuing calibration.

10.6.3.1. If none of the CCCs are required analytes, project specific calibration specifications must be agreed with the client.

10.6.3.2. For Method 8270D, any sample non-detects for an analyte that fails the SOP criteria low, must have a low level CCV (CCV at the RL) in the batch as a sensitivity demonstration. The criterion for a passing LLCCV is detection only, and a passing LLCCV allows non-detect samples to be reported without flagging.

10.6.4. Once the above criteria have been met, sample analysis will begin. Initial calibration average RFs (or the calibration curve) will be used for sample quantitation, not the continuing calibration RFs. Analysis will proceed until 12 hours from the injection of the DFTPP have passed. (A sample *injected* less than 12 hours after the DFTPP is acceptable.)

11. PROCEDURE

11.1 Sample Preparation

11.1.1 Samples are prepared following SOP NC-OP-032.

11.1.2 For DoD work, refer to SOP NC-QA-016 for specific details.

11.2 Sample Analysis Procedure

11.2.1 Calibrate the instrument as described in Section 10. Depending on the target compounds required by the client, it may be necessary to use more than one calibration standard.

11.2.2 Analyze all samples using the same instrument conditions as the preceding continuing calibration standard.

11.2.3 Add internal standard to the extract to result in 2 ng injected on column. Mix thoroughly before injection into the instrument.

11.2.4 Inject the sample extract into the GC/MS system using the same injection technique as used for the standards.

11.2.5 The data system will determine the concentration of each analyte in the extract using calculations equivalent to those in Section 12. Quantitation is based on the initial calibration, not the continuing calibration.

11.2.6 Identified compounds are reviewed for proper integration. Manual integrations are performed if necessary and are documented by the analyst or automatically by the data system. Chromatograms before and after manual integration are required by many programs. Additional information on manual integration can be found in SOP CA-Q-S-002.

11.2.7 Target compounds identified by the data system are evaluated using the criteria listed in Section 12.1.

11.2.8 Library searches of peaks present in the chromatogram that are not target compounds (Tentatively Identified Compounds, TIC) must be performed if required by the client. They are evaluated using the criteria in Section 12.3.

11.3 Dilutions

11.3.1 If the response for any compound exceeds the working range of the GC/MS system, a dilution of the extract is prepared and analyzed. An appropriate dilution must be in the upper half of the calibration range. Samples must be screened to determine the appropriate dilution for the initial run. If the initial diluted run has no hits or hits below 20% of the calibration range and the matrix allows for analysis at a lesser dilution, the sample must be re-analyzed at a dilution targeted to bring the largest hit above 50% of the calibration range.

11.3.2 Guidance for Dilutions Due to Matrix

11.3.2.1 If the sample is initially run at a dilution and the baseline rise is less than the height of the internal standards, or if individual non-target peaks are less than two times the height of the internal standards, the sample should be re-analyzed at a more concentrated dilution. This requirement is approximate and subject to analyst judgement. For example, samples containing organic acids must be analyzed at a higher dilution to avoid destroying the column.

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11.3.3 Reporting Dilutions

11.3.3.1 The most concentrated dilution with target compounds within the calibration range will be reported. Other dilutions will only be reported at client request.

11.4 Perform all qualitative and quantitative measurements. When the extracts are not being used for analyses, refrigerate them at 4 $\pm$2^{o}C protected from light in screw cap vials equipped with unpierced Teflon®-lined septa.

11.5 Retention Time Criteria for Samples

11.5.1 If the retention time for any internal standard changes by more than 0.5 minutes from the last continuing calibration standard, the chromatographic system must be inspected for malfunctions and corrected. Re-analysis of samples analyzed while the system was malfunctioning is required.

11.5.2 If the retention time of any internal standard in any sample varies by more than 0.1 minute from the preceding continuing calibration standard, the data must be carefully evaluated to ensure no analytes have shifted outside their retention time windows.

11.6 Procedural Variations

11.6.1 One-time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, chemistry, sample size, or other parameters. Any variation in procedure must be completely documented using a Nonconformance Memo and approved by a Technical Specialist and QA Manager. The Nonconformance Memo must be filed in the project file. Any unauthorized deviations from this procedure must also be documented as a non-conformance with a cause and corrective action described.

11.7 Troubleshooting Guide

11.7.1 Daily Instrument Maintenance

11.7.1.1 In addition to the checks listed in the instrument maintenance schedule in the TestAmerica North Canton Quality Assurance Manual (QAM), current version, the following daily maintenance must be performed.

11.7.1.1.1 Clip column as necessary.

11.7.1.1.2 Install new or cleaned injection port liner as necessary.

11.7.1.1.3 Install new septum as necessary.

11.7.1.1.4 Perform autotune.

11.7.2 Major Maintenance

11.7.2.1 A new initial calibration is necessary following major maintenance. Major maintenance includes changing the column, cleaning the source, and replacing the multiplier. Refer to the manufacturer's manual for specific guidance.

12. DATA ANALYSIS AND CALCULATIONS

12.1 Qualitative Identification

12.1.1 An analyte is identified by retention time and by comparison of the sample mass spectrum with the mass spectrum of a standard of the suspected compound (standard reference spectrum). Mass spectra for standard reference may be obtained on the user's GC/MS by analysis of the calibration standards or from the NBS library. Two criteria must be satisfied to verify identification: (1) elution of sample component at the same GC retention time as the standard component; and (2) correspondence of the sample component and the standard component characteristic ions. (Note: Care must be taken to ensure that spectral distortion due to co-elution is evaluated.)

12.1.1.1 The sample component retention time must compare to within ± 0.2 min. of the retention time of the standard component. For reference, the standard must be run within the same 12 hours as the sample.

12.1.1.2 All ions present in the standard mass spectra at a relative intensity greater than 10% (most abundant ion in the spectrum equals 100%) must be present in the sample spectrum.

12.1.1.3 The characteristic ions of a compound must maximize in the same scan or within one scan of each other.

12.1.1.4 The relative intensities of ions must agree to within ±30% between the standard and sample spectra. (Example: For an ion with an abundance of 50% in the standard spectra, the corresponding sample abundance must be between 20% and 80%.)

12.1.2 If a compound cannot be verified by all the above criteria, but in the technical judgment of the analyst the identification is correct, the analyst must report that identification and proceed with quantitation.

12.2 Mass chromatogram searches

12.2.1 Certain compounds are unstable in the calibration standard and cannot be calibrated in the normal way. In particular, the compound hexachlorophene (CAS 70-30-4) falls into this category, and is required for Appendix IX analysis. For this analyte, a mass chromatogram search is made.

12.2.1.1 Hexachlorophene

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12.2.1.1.1 Display the mass chromatograms for mass 196 and mass 198 for the region of the chromatogram from at least 2 minutes before chrysene-d12 to at least 4 minutes after chrysene-d12. If peaks for both ions coincide, then the analyst evaluates the spectrum for the presence of hexachlorophene. No quantitation is possible.

12.3 For samples containing components not associated with the calibration standards, a library search may be made for the purpose of tentative identification. The necessity to perform this type of identification must be determined by the type of analyses being conducted or by client request. Computer-generated library search routines must not use normalization routines that would misrepresent the library or unknown spectra when compared to each other. Only after visual comparison of sample spectra with the nearest library searches will the mass spectral interpretation specialist assign a tentative identification. Guidelines for making tentative identification are:

12.3.1 Relative intensities of major ions in the reference spectrum (ions >10% of the most abundant ion) must be present in the sample spectrum.

12.3.2 The relative intensities of the major ions must agree within ±20%. (Example: For an ion with an abundance of 50% in the standard spectrum, the corresponding sample ion abundance must be between 30%and 70%.)

12.3.3 Molecular ions present in the reference spectrum must be present in the sample spectrum.

12.3.4 Ions present in the sample spectrum, but not in the reference spectrum, must be reviewed for possible background contamination or presence of co-eluting compounds.

12.3.5 Ions present in the reference spectrum, but not in the sample spectrum, should be reviewed for possible subtraction from the sample spectrum because of background contamination or co-eluting peaks. Data system library reduction programs can sometimes create these discrepancies.

12.3.6 Automatic background subtraction can severely distort spectra from samples with unresolved hydrocarbons.

12.3.7 Note: For water samples, the TIC searches begin with compounds eluting after the first surrogate (2-Fluorophenol). For solid samples, the TIC searches begin with compounds eluting after the Aldol Condensation Product. Any compounds eluting before these analytes are considered volatile analytes are reported in the volatile analysis. A possible exception to this general rule would be if an early eluting compound were the reason for a sample dilution.

12.3.8 If a client requests 10 TICs, the laboratory supplies a minimum of 10. For a request of 20 TICS, the laboratory would supply a minimum of 20--assuming that number of compounds were available.

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12.4 Anyone evaluating data is trained to know how to handle isomers with identical mass spectra and close elution times. These include:

Dichlorobenzenes
Methylphenols
Trichlorophenols
Phenanthrene, anthracene
Fluoranthene, pyrene
Benzo(b) and (k)fluoranthene
Chrysene, benzo(a)anthracene

Extra precautions concerning these compounds are to more closely scrutinize retention time vs. the calibration standard and also to check that all isomers have distinct retention times.

A second category of problem compounds would be the poor responders or compounds that chromatograph poorly. Included in this category would be:

Benzoic acid
Chloroanilines
Nitroanilines
2,4-Dinitrophenol
4-Nitrophenol
Pentachlorophenol
3,3'-Dichlorobenzidine
Benzyl alcohol
4,6-Dinitro-2-methylphenol

Manually checking the integrations would be appropriate for these compounds.

12.5 Calculations

12.5.1 Percent Relative Standard Deviation for Initial Calibration

$$\%RSD = \frac{SD}{\overline{RF}} \times 100$$

$RF =$ Mean of RFs from intial caibration for a compound

$SD =$ Standard deviation of RFs from initial calibration for a compound,

$$= \sqrt{\sum_{i=1}^{N} \frac{\left(RFi - \overline{RF}\right)^2}{N-1}}$$

$RFi =$ RF for each of the calibration levels

$N =$ Number of RF values

12.5.2 Continuing calibration percent drift

$$\%Drift = \frac{C_{actual} - C_{found}}{C_{actual}} \times 100\%$$

C_{actual} = Known concentration in standard

C_{found} = Measured concentration using selected quantitation method

12.5.3 Concentration in the extract

The concentration of each identified analyte and surrogate in the extract is calculated from the linear or quadratic curve fitted to the initial calibration points, or from the average RF of the initial calibration.

12.5.3.1 Average Response Factor

If the average of all the %RSDs of the response factors in the initial calibration is $\leq$ 15%, the average response factor from the initial calibration may be used for quantitation.

$$C_{ex} = \frac{R_x C_{is}}{R_{is}\overline{RF}}$$

12.5.3.2 Linear fit

$$C_{ex} = A + B\frac{(R_x C_{is})}{R_{is}}$$

Where: C_{ex} = Concentration in extract, µg/mL
R_x = Response for analyte
C_{is} = Concentration of internal standard
A = Intercept
B = Slope

12.5.3.3 Quadratic fit

$$C_{ex} = A + B\left(\frac{R_x C_{is}}{R_{is}}\right) + C\left(\frac{R_x C_{is}}{R_{is}}^2\right)$$

Where: C = Curvature

12.5.4 The concentration in the sample is then calculated.

12.5.4.1 Aqueous Calculation

$$\text{Concentration, } \mu\text{g / L} = \frac{C_{ex} V_t}{V_o}$$

Where: V_t = Volume of total extract, µL, taking into account dilutions (i.e., a 1-to-10 dilution of a 1 Ml extract will mean V_t = 10,000 µL. If half the base/neutral extract and half the acid extract are combined, V_t = 2,000.)

V_o = Volume of water extracted (mL)

12.5.5 Sediment/Soil, Sludge (on a dry-weight basis) and Waste (normally on a wet-weight basis

$$\text{Concentration, } \mu\text{g / kg} = \frac{C_{ex} V_t}{W_s D}$$

Where: W_s = Weight of sample extracted or diluted in grams
D = (100 - % moisture in sample)/100, for a dry weight basis or one for a wet weight basis

12.6 MS/MSD percent recovery calculation.

$$\text{Matrix Spike Recovery} = \frac{S_{SR} - S_R}{S_A} \times 100\%$$

Where: S_{SR} = Spike sample result
S_R = Sample result
S_A = Concentration equivalent of spike added

12.7 Relative % Difference calculation for the MS/MSD

$$RPD = \frac{MS_R - MSD_R}{1/2(MS_R + MSD_R)} \times 100$$

Where: RPD = Relative percent difference
MS_R = Matrix spike result
MSD_R = Matrix spike duplicate result

12.8 Relative response factor calculation

$$RF = \frac{A_x C_{is}}{A_{is} C_x}$$

Where: A_x = Area of the characteristic ion for the compound being measured
A_{is} = Area of the characteristic ion for the specific internal standard
C_x = Concentration of the compound being measured (µg/L)
C_{is} = Concentration of the specific internal standard (µg/L)

12.9 Calculation of TICs: The calculation of TICs (tentatively identified compounds) is identical to the above calculations with the following exceptions:

A_x = Area of the total ion chromatogram for the compound being measured
A_{is} = Area of the total ion chromatogram for the nearest internal standard without interference
RF = 1

12.10 Percent DDT breakdown

$$\% \text{ DDT breakdown} = \frac{\text{DDEarea} + \text{DDDarea}}{\text{DDTarea} + \text{DDEarea} + \text{DDarea}}$$

The total ion current areas are used for this calculation

12.11 Additional equations and calculations are listed in the following SOPs: Calibration Curves (General), CA-Q-S-005, and Selection of Calibration Points, CA-T-P-002

13. METHOD PERFORMANCE

13.1 Method Detection Limit

13.1.1 Each laboratory must generate a valid method detection limit for each analyte of interest. The MDL must be below the reporting limit for each analyte. The procedure for determination of the method detection limit is given in 40 CFR Part 136, Appendix B, and further defined in Policy CA-Q-S-006 and SOP NC-QA-021.

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13.2 Initial Demonstration

13.2.1 Each laboratory must make an initial demonstration of capability for each individual method. Demonstration of capability for both soil and water matrices is required. This requires analysis of Laboratory Control Samples (LCS) containing all of the standard analytes for the method. For some tests, it may be necessary to use more than one QC check mix to cover all analytes of interest.

13.2.1.1 Four aliquots of the LCS are analyzed using the same procedures used to analyze samples, including sample preparation.

13.2.1.2 Calculate the average recovery and standard deviation of the recovery for each analyte of interest.

13.2.1.3 If any analyte does not meet the LCS acceptance criteria the test must be repeated. Only those analytes that did not meet criteria in the first test need to be evaluated. Repeated failure for any analyte indicates the need for the laboratory to evaluate the analytical procedure and take corrective action.

13.3 Training Qualification

13.3.1 The Group/Team Leader has the responsibility to ensure this procedure is performed by an analyst who has been properly trained in its use and has the required experience.

13.3.2 Method validation information (where applicable) in the form of laboratory demonstrations of capabilities is maintained for this method in the laboratory QA files.

14. POLLUTION PREVENTION

14.1 This section is not applicable to this procedure.

15. WASTE MANAGEMENT

15.1 All waste will be disposed of in accordance with Federal, State and Local laws and regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention."

15.2 Laboratory personnel assigned to perform hazardous waste disposal procedures must have a working knowledge of the established procedures and practices of TestAmerica. They must have training on the hazardous waste disposal practices upon initial assignment to these tasks, followed by annual refresher training.

15.3 Waste Streams Produced by the Method

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15.3.1 Vials containing sample extracts. These vials are placed in the vial waste located in the GC/MS laboratory.

16. REFERENCES

16.1 References

16.1.1 SW846, Test Methods for Evaluating Solid Waste, Third Edition, Update III October 1994, Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS): Capillary Column Technique, Method 8270C

16.1.2 J. W. Eichelberger, L. E. Harris, and W. L. Budde, "Reference Compound to Calibrate Ion Abundance Measurement in Gas Chromatography/Mass Spectrometry," Analytical Chemistry, 47, 995 (1975)

16.1.3 TestAmerica North Canton Quality Assurance Manual (QAM), current version

16.1.4 TestAmerica Corporate Environmental Health and Safety Manual, CW-E-M-001, and TestAmerica North Canton Facility Addendum and Contingency Plan, current version

16.1.5 Corporate Quality Management Plan (CQMP), current version

16.1.6 Revision History

Historical File:		Revision 2.1: 01/25/99		Revision 0: 05/28/08 (NC-MS-018)
(formerly CORP-MS-0001NC)		Revision 2.2: 03/27/00		Revision 1: 12/16/08
		Revision 2.3: 02/15/01		
		Revision 2.4: 05/29/01		
		Revision 2.5: 04/25/02		
		Revision 2.6: 08/15/02		
		Revision 2.7: 11/12/02		
		Revision 2.8: 01/23/03		
		Revision 2.9: 06/18/03		
		Revision 2.10: 02/24/04		
		Revision 2.11: 02/03/06		
		Revision 2.12: 03/01/07		

16.2 Associated SOPs and policies, current version

16.2.1 QA Policy, QA-003

16.2.2 Statistical Evaluation of Data and Development of Control Charts, NC-QA-018

16.2.3 Method Detection Limits and Instrument Detection Limits, NC-QA-021 and CA-Q-S-006

16.2.4 Supplemental Practices for DoD Project Work, NC-QA-016

16.2.5 Standard and Reagents, NC-QA-017

16.2.6 Acceptable Manual Integration Practices, CA-Q-S-002

16.2.7 Calibration Curves (General), CA-Q-S-005

16.2.8 Section of Calibration Points, CA-T-P-002

17. MISCELLANEOUS

17.1 Modifications from Reference Method

17.1.1 A retention time window of 0.2 minutes is used for all components, since some data systems do not have the capability of using the relative retention time units specified in the reference method.

17.1.2 The quantitation and qualifier ions from compounds have been changed from those recommended in SW-846 in order to improve the reliability of qualitative identification.

17.2 Tables

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TABLE 1: TestAmerica North Canton Standard Reporting Limits

Analytes	CAS Number	Water µg/L	Soil µg/kg	Low Level Water, µg/L	Low Level Soil, µg/kg	TCLP mg/L
1,1-Biphenyl	92-52-4	10	330	1	50	
1,2,4-Trichlorobenzene	120-82-1	10	330	1	50	
1,2-Dichlorobenzene	95-50-1	10	330	1	50	
1,3-Dichlorobenzene	541-73-1	10	330	1	50	
1,4-Dichlorobenzene	106-46-7	10	330	1	50	0.004
1-Methyl Naphthalene	90-12-0	10	330	0.2	6.67	
2,2'-oxybis(1-chloropropane)[1]	108-60-1	10	330	1	100	
2,4,5-Trichlorophenol	95-95-4	10	330	5	150	0.02
2,4,6-Trichlorophenol	88-06-2	10	330	5	150	0.02
2,4-Dichlorophenol	120-83-2	10	330	2	150	
2,4-Dimethylphenol	105-67-9	10	330	2	150	
2,4-Dinitrophenol	51-28-5	50	1600	5	330	
2,4-Dinitrotoluene	121-14-2	10	330	5	200	0.02
2,6-Dinitrotoluene	606-20-2	10	330	5	200	
2-Chloronaphthalene	91-58-7	10	330	1	50	
2-Chlorophenol	95-57-8	10	330	1	50	
2-Methylnaphthalene	91-57-6	10	330	0.2	6.67	
2-Methylphenol	95-48-7	10	330	1	200	
2-Nitroaniline	88-74-4	50	1600	2	200	
2-Nitrophenol	88-75-5	10	330	2	50	
3,3'-Dichlorobenzidine	91-94-1	50	1600	5	100	
3-Nitroaniline	99-09-2	50	1600	2	200	
4,6-Dinitro-2-methylphenol	534-52-1	50	1600	5	150	
4-Bromophenyl phenyl ether	101-55-3	10	330	2	50	
4-Chloro-3-methylphenol	59-50-7	10	330	2	150	
4-Chloroaniline	106-47-8	10	330	2	150	
4-Chlorophenyl phenyl ether	7005-72-3	10	330	2	50	
4-Methylphenol	106-44-5	10	330	1	200	
4-Nitroaniline	100-01-6	50	1600	2	200	
4-Nitrophenol	100-02-7	50	1600	5	330	
Acenaphthene	83-32-9	10	330	0.2	6.67	
Acenaphthylene	208-96-8	10	330	0.2	6.67	
Aniline	62-53-3	10	330	5	330	
Anthracene	120-12-7	10	330	0.2	6.67	
Atrazine	1912-24-9	10	330	1	200	
Azobenzene	103-33-3	10	330	10	330	
Benzaldehyde	100-52-7	10	330	1	100	
Benzenethiol	108-98-5	10	330	10	330	
Benzidine	92-87-5	100	3300	5	660	
Benzo(a)anthracene	56-55-3	10	330	0.2	6.67	
Benzo(a)pyrene	50-32-8	10	330	0.2	6.67	
Benzo(b)fluoranthene	205-99-2	10	330	0.2	6.67	
Benzo(g,h,i)perylene	191-24-2	10	330	0.2	6.67	
Benzo(k)fluoranthene	207-08-9	10	330	0.2	6.67	

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Analytes	CAS Number	Water µg/L	Soil µg/kg	Low Level Water, µg/L	Low Level Soil, µg/kg	TCLP mg/L
Benzoic acid	65-85-0	50	1600	25	660	
Benzyl alcohol	100-51-6	10	330	5	330	
Bis(2-chloroethoxy)methane	111-91-1	10	330	1	100	
Bis(2-chloroethyl)ether	111-44-4	10	330	1	100	
Bis(2-ethylhexyl)phthalate	117-81-7	10	330	2	50	
Butyl benzyl phthalate	85-68-7	10	330	1	50	
Caprolactam	105-60-2	10	330	5	330	
Carbazole	86-74-8	10	330	1	50	
Chrysene	218-01-9	10	330	0.2	6.67	
Dibenz(a,h)anthracene	53-70-3	10	330	0.2	6.67	
Dibenzofuran	132-64-9	10	330	1	50	
Diethylphthalate	84-66-2	10	330	1	50	
Dimethyl phthalate	131-11-3	10	330	1	50	
Di-n-butyl phthalate	84-74-2	10	330	1	50	
Di-n-octylphthalate	117-84-0	10	330	1	50	
Fluoranthene	206-44-0	10	330	0.2	6.67	
Fluorene	86-73-7	10	330	0.2	6.67	
Hexachlorobenzene	118-74-1	10	330	0.2	6.67	0.02
Hexachlorobutadiene	87-68-3	10	330	1	50	0.02
Hexachlorocyclopentadiene	77-47-4	10	1600	10	330	0.05
Hexachloroethane	67-72-1	10	330	1	50	
Indene	95-13-6	10	330	5	330	
Indeno(1,2,3-cd)pyrene	193-39-5	10	330	0.2	6.67	
Isophorone	78-59-1	10	330	1	50	
Naphthalene	91-20-3	10	330	0.2	6.67	
Nitrobenzene	98-95-3	10	330	1	100	0.004
N-nitrosodimethylamine	62-75-9	10	330	1	100	
N-Nitroso-di-n-propylamine	621-64-7	10	330	1	50	
N-Nitrosodiphenylamine	86-30-6	10	330	1	50	
Pentachlorophenol	87-86-5	10	330	5	150	0.04
Phenanthrene	85-01-8	10	330	0.2	6.67	
Phenol	108-95-2	10	330	1	50	
Pyrene	129-00-0	10	330	0.2	6.67	
Pyridine	110-86-1	20	660	1	100	0.02
Quinoline	91-22-5	10	330	5	330	
m-Cresol & p Cresol						0.04
o-Cresol						0.004
1,2,4,5-Tetrachlorobenzene	95-94-3	10	330	1	100	
1,3,5-Trinitrobenzene	99-35-4	50	1600	5	1600	
1,3-Dinitrobenzene	99-65-0	10	330	2	330	
1,4-Dinitrobenzene	100-25-4	10	330	2	100	
1,4-Naphthoquinone	130-15-4	50	1600	50	330	
1-Naphthylamine	134-32-7	10	330	2	330	
2,3,4,6-Tetrachlorophenol	58-90-2	50	1600	10	100	
2,6-Dichlorophenol	87-65-0	10	330	5	150	
2-Acetylaminofluorene	53-96-3	100	3300	10	330	

Analytes	CAS Number	Water µg/L	Soil µg/kg	Low Level Water, µg/L	Low Level Soil, µg/kg	TCLP mg/L
2-Naphthylamine	91-59-8	10	330	2	200	
2-Picoline	109-06-8	20	660	5	330	
2-secbutyl-4,6-dinitrophenol (Dinoseb2)	88-85-7	20	660	2	330	
3,3'-Dimethylbenzidine	119-93-7	50	1600	5	330	
3-Methylcholanthrene	56-49-5	20	660	5	200	
3-Methylphenol	108-39-4	10	330	1	200	
4-Aminobiphenyl	92-67-1	50	1600	5	330	
4-Nitroquinoline-1-oxide	56-57-5	100	3300	5	330	
5-Nitro-o-toluidine	99-55-8	20	660	2	330	
7,12-Dimethylbenz(a)anthracene	57-97-6	20	660	2	330	
a,a-Dimethyl-phenethylamine	122-09-8	50	1600	5	660	
Acetophenone	98-86-2	10	330	1	100	
Aramite	140-57-8	20	660	5	330	
Diallate[2]	2303-16-4	20	660	10	330	
Dibenz(a,j)acridine	224-42-0	20	660	5	330	
Dimethoate	60-51-5	20	660	2	330	
Disulfoton	298-04-4	50	1600	2	330	
Ethyl methanesulfonate	62-50-0	10	330	2	330	
Famphur	52-85-7	100	3300	10	3300	
Hexachloropropene	1888-71-7	100	3300	5	0.02	
Isosafrole	120-58-1	20	660	5	330	
Methapyrilene	91-80-5	50	1600	2	330	
Methyl methanesulfonate	66-27-3	10	330	2	330	
N-Nitrosodiethylamine	55-18-5	10	330	2	100	
n-Nitrosodi-n-butylamine	924-16-3	10	330	2	100	
N-Nitrosomethylethylamine	10595-95-6	10	330	2	100	
N-Nitrosomorpholine	59-89-2	10	330	2	330	
N-Nitrosopiperidine	100-75-4	10	330	2	330	
N-Nitrosopyrrolidine	930-55-2	10	330	2	50	
o,o,o-Triethyl-Phosphorothioate	126-68-1	50	1600	2	330	
o-Toluidine	95-53-4	20	660	2	330	
p-(Dimethylamino)azobenzene	60-11-7	20	660	2	330	
p-Chlorobenzilate	510-15-6	10	330	2	330	
Pentachlorobenzene	608-93-5	10	330	2	100	
Pentachloroethane	76-01-7	50	1600	20	330	
Pentachloronitrobenzene	82-68-8	50	1600	2	330	
Phenacetin	62-44-2	20	660	2	330	
Phorate	298-02-2	50	1600	2	330	
p-Phenylenediamine	106-50-3	100	3300	40	660	
Pronamide	23950-58-5	20	660	2	330	
Safrole	94-59-7	20	660	2	330	
Sulfotepp	3689-24-5	50	1600	5	330	
Thionazin	297-97-2	50	1600	2	330	
1,2,4,5-Tetrachlorobenzene	95-94-3	10	330	1	100	
1,3,5-Trinitrobenzene	99-35-4	50	1600	5	1600	
1,3-Dinitrobenzene	99-65-0	10	330	2	330	

Analytes	CAS Number	Water µg/L	Soil µg/kg	Low Level Water, µg/L	Low Level Soil, µg/kg	TCLP mg/L
1,4-Dinitrobenzene	100-25-4	10	330	2	100	

1 2,2'oxybis(1-chloropropane) was formerly known as bis (2-chloroisopropyl)ether.

Skinner List Compound

Hexachlorophene is a required analyte for Appendix IX. This compound is not stable, and therefore not included in the calibration standard. The characteristic ions for hexachlorophene are searched for in the chromatogram (see Section 12.2.1).

Diphenylamine is a required compound for Appendix IX. N-nitrosodiphenylamine decomposes in the injection port to form diphenylamine. Therefore, these two compounds cannot be distinguished. Diphenylamine is not included in the calibration standard.

TABLE 2: TestAmerica North Canton Michigan Program[1]

Semivolatile	CAS Number	Michigan Reporting Limits	
		Aqueous mg/L	Low Soil/Sediment mg/kg
Acenaphthene	83-32-9	5	330
Acenaphthylene	208-96-8	5	330
Acetophenone	98-86-2	5	330
Anthracene	120-12-7	5	330
Atrzine	1912-24-9	5	330
Benzaldehyde	100-52-7	10	330
Benzo(a)anthracene	56-55-3	1	330
Benzo(a)pyrene	50-32-8	2	330
Benzo(b)fluoranthene	205-99-2	2	330
Benzo(g,h,i)perylene	191-24-2	5	330
Benzo(k)fluoranthene	207-08-9	5	330
1,1'-Biphenyl	92-52-4	10	330
4-Bromophenylphenyl ehter	101-55-3	5	330
Butylbenzylphthalate	85-68-7	5	330
di-n-Butylphthalate	84-74-2	5	330
Caprolactam	105-60-2	10	330
Carbazole	86-74-8	10	330
4-Chloroaniline	106-47-8	20	1700
bis(2-Chloroethoxy)methane	111-91-1	5	330
bis(2-Chloroethyl)ether	111-44-4	4	330
bis(2-Chloroisopropyl)ether	108-60-1	5	330
4-Chloro-3-Methylphenol	59-50-7	5	330
2-Chloronaphthalene	91-58-7	5	330
2-Chlorophenol	95-57-8	5	330
4-Chlorophenyl phenyl ether	7005-72-3	5	330
Chrysene	218-01-9	5	330

		Michigan Reporting Limits	
Dibenz(a,h)anthracene	53-70-3	2	330
Dibenzofuran	132-64-9	5	330
3,3'-Dichlorobenzidine	91-94-1	4	2000
2,4-Dichlorophenol	120-83-2	10	330
Diethylphthalate	84-66-2	5	330
2-4-Dimethylphenol	105-67-9	5	330
Dimethylphthalate	131-11-3	5	330

Semivolatile	**CAS Number**	**Michigan Reporting Limits**	
		Aqueous µg/L	**Low Soil/Sediment µg/kg**
4,6-Dinitro-2-methylphenol	534-52-1	20	1700
2,4-Dinitrophenol	51-28-5	20	1700
2,4-Dinitrotoluene	121-14-2	5	330
2,6-Dinitrotoluene	606-20-2	5	330
bis(2-Ethylhexyl)phthalate	117-81-7	5	330
Fluoranthene	206-44-0	5	330
Fluorene	86-73-7	5	330
Hexachlorobenzene	118-74-1	5	330
Hexachlorobutadiene	87-68-3	5	330
Hexachlorocyclopentadiene	77-47-4	5	330
Hexachloroethane	67-72-1	5	330
Indeno(1,2,3-cd)pyrene	193-39-5	2	330
Isophorone	78-59-1	5	330
2-Methylnapthalene	91-57-6	5	330
2-Methylphenol	95-48-7	5	330
4-Methylphenol	106-44-5	5	330
Naphthalene	91-20-3	5	330
2-Nitroaniline	88-74-4	20	1700
3-Nitroaniline	99-09-2	20	1700
4-Nitroaniline	100-01-6	20	1700
Nitrobenzene	95-95-3	4	330
2-Nitrophenol	88-75-5	5	330
4-Nitrophenol	100-02-7	20	1700
N-Nitroso-di-n-propylamine	621-64-7	5	330
N-Nitrosodiphenylamine (diphenylamine)	62-75-9	5	330
di-n-Octylphthalate	117-84-0	5	330
Pentachlorophenol	87-86-5	20	800
Phenanthrene	85-01-8	5	330
Phenol	108-95-2	5	330
Pyrene	129-00-0	5	330
2,4,5-Trichlorophenol	95-95-4	5	330
2,4,6-Trichlorophenol	88-06-2	4	330

[1] Reporting Limits are only for samples performed under the Michigan program.

TABLE 3: Suggested Instrument Conditions

Mass Range	35-500 amu
Scan Time	≤1 second/scan
Initial Column Temperature/Hold Time	60°C for 1 minutes
Column Temperature Program	60 - 320°C at 35°C/min for 3 min
Final Column Temperature/Hold Time	320°C (until at least one minute after benzo(g,h,i)perylene has eluted)
Injector Temperature	250 - 300°C
Transfer Line Temperature	250 - 300°C
Source Temperature	According to manufacturer's Specifications
Injector	Grob-type, split / splitless
Sample Volume	0.5 µl
Carrier Gas	Helium at 30 cm/sec

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TABLE 4: DFTPP Key Ions and Ion Abundance Criteria

Mass	Ion Abundance Criteria
51	30 – 80% of mass 198
68	<2% of mass 69
70	<2% of mass 69
127	25 - 75% of mass 198
197	<1% of mass 198
198	Base peak, 100% relative abundance
199	5 – 9% of mass 198
275	10 – 30% of mass 198
365	> 0.75% of mass 198
441	Present, but less than mass 443
442	40 - 110% of mass 198
443	15 - 24% of mass 442

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TABLE 5: Analytes in Approximate Retention Time Order and Characteristic Ions

Analyte	Primary	Secondary	Tertiary
N-nitrosodimethylamine	74	42	
Pyridine	79	52	
2-Fluorophenol (Surrogate Standard)	112	64	63
Phenol-d5 (Surrogate Standard)	99	42	71
Benzaldehyde	77	105	106
Aniline	93	66	
Phenol	94	65	66
Bis(2-chloroethyl)ether	93	63	95
2-Chlorophenol	128	64	130
1,3-Dichlorobenzene	146	148	113
1,4-Dichlorobenzene-d4 (Internal Standard)	152	150	115
1,4-Dichlorobenzene	146	148	113
Benzyl Alcohol	108	79	77
1,2-Dichlorobenzene	146	148	113
2-Methylphenol	108	107	79
2,2'-oxybis(1-chloropropane)[1]	45	77	79
4-Methylphenol	108	107	79
N-Nitroso-di-n-propylamine	70	42	101,130
Hexachloroethane	117	201	199
Nitrobenzene-d5 (Surrogate Standard)	82	128	54
Nitrobenzene	77	123	65
Isophorone	82	95	138
2-Nitrophenol	139	65	109
2,4-Dimethylphenol	107	121	122
Benzoic Acid	122	105	77
Bis(2-chloroethoxy)methane	93	95	123
2,4-Dichlorophenol	162	164	98
1,2,4-Trichlorobenzene	180	182	145
Naphthalene-d8 (Internal Standard)	136	68	54
Naphthalene	128	129	127
4-Chloroaniline	127	129	65
Hexachlorobutadiene	225	223	227
Caprolactam	113	55	56
4-Chloro-3-methylphenol	107	144	142
2-Methylnaphthalene	142	141	115
Hexachlorocyclopentadiene	237	235	272
2,4,6-Trichlorophenol	196	198	200
2,4,5-Trichlorophenol	196	198	200
1,1'-Biphenyl	154	153	76
2-Fluorobiphenyl (Surrogate Standard)	172	171	170
2-Chloronaphthalene	162	164	127
2-Nitroaniline	65	92	138
Dimethylphthalate	163	194	164
Acenaphthylene	152	151	153

TABLE 5: Analytes in Approximate Retention Time Order and Characteristic Ions

Analyte	Primary	Secondary	Tertiary
2,6-Dinitrotoluene	165	63	89

Analyte	Primary	Secondary	Tertiary
Acenaphthene-d10 (Internal Standard)	164	162	160
3-Nitroaniline	138	108	92
Acenaphthene	153	152	154
2,4-Dinitrophenol	184	63	154
Dibenzofuran	168	139	84
4-Nitrophenol	109	139	65
2,4-Dinitrotoluene	165	63	89
Diethylphthalate	149	177	150
Fluorene	166	165	167
4-Chlorophenylphenylether	204	206	141
4-Nitroaniline	138	92	108
4,6-Dinitro-2-methylphenol	198	182	77
N-Nitrosodiphenylamine	169	168	167
2,4,6-Tribromophenol (Surrogate Standard)	330	332	141
Azobenzene	77	182	105
4-Bromophenylphenylether	248	250	141
Hexachlorobenzene	284	142	249
Atrazine	200	173	215
Pentachlorophenol	266	264	268
Phenanthrene-d10 (Internal Standard)	188	94	80
Phenanthrene	178	179	176
Anthracene	178	179	176
Carbazole	167	166	139
Di-n-butylphthalate	149	150	104
Fluoranthene	202	101	100
Benzidine	184	92	185
Pyrene	202	101	100
Terphenyl-d14 (Surrogate Standard)	244	122	212
Butylbenzylphthalate	149	91	206
Benzo(a)Anthracene	228	229	226
Chrysene-d12 (Internal Standard)	240	120	236
3,3'-Dichlorobenzidine	252	254	126
Chrysene	228	226	229
Bis(2-ethylhexyl)phthalate	149	167	279
Di-n-octylphthalate	149	167	43
Benzo(b)fluoranthene	252	253	125
Benzo(k)fluoranthene	252	253	125
Benzo(a)pyrene	252	253	125
Perylene-d12 (Internal Standard)	264	260	265
Indeno(1,2,3-cd)pyrene	276	138	277
Dibenz(a,h)anthracene	278	139	279
Benzo(g,h,i)perylene	276	138	277
2-Picoline	93	66	92

Analyte	Primary	Secondary	Tertiary
N-Nitrosomethylethylamine	88	42	43
Methyl methanesulfonate	80	79	65
N-Nitrosodiethylamine	102	44	57
Ethyl methanesulfonate	79	109	97
Pentachloroethane	117	119	167
Acetophenone	105	77	120
N-Nitrosopyrrolidine	100	41	42
N-Nitrosomorpholine	116	56	86
o-Toluidine	106	107	
3-Methylphenol	108	107	77
N-Nitrosopiperidine	114	42	55
o,o,o-Triethyl-Phosphorothioate	198	121	93
a,a-Dimethyl-phenethylamine	58	91	
2,6-Dichlorophenol	162	164	63
Hexachloropropene	213	215	211
p-Phenylenediamine	108	80	
n-Nitrosodi-n-butylamine	84	57	41
Safrole	162	104	77
1,2,4,5-Tetrachlorobenzene	216	214	218
Isosafrole 1	162	104	131
Isosafrole 2	162	104	131
1,4-Dinitrobenzene	168	75	122
1,4-Naphthoquinone	158	104	102
1,3-Dinitrobenzene	168	75	76
Pentachlorobenzene	250	248	252
1-Naphthylamine	143	115	
2-Naphthylamine	143	115	
2,3,4,6-Tetrachlorophenol	232	230	131
5-Nitro-o-toluidine	152	77	106
Thionazin	97	96	143
1,3,5-Trinitrobenzene	213	75	120
Sulfotepp	97	322	202
Phorate	75	97	121
Phenacetin	108	179	109
Diallate	86	234	
Dimethoate	87	93	125
4-Aminobiphenyl	169		
Pentachloronitrobenzene	237	142	214
Pronamide	173	175	255
Disulfoton	88	97	89
2-secbutyl-4,6-dinitrophenol (Dinoseb)	211	163	147
Methyl parathion	109	125	263
4-Nitroquinoline-1-oxide	190	128	160
Famphur	218	125	93
Methapyrilene	97	58	
Aramite 1	185	319	
Aramite 2	185	319	
p-(Dimethylamino)azobenzene	120	225	77

Analyte	Primary	Secondary	Tertiary
p-Chlorobenzilate	251	139	253
3,3'-Dimethylbenzidine	212	106	
2-Acetylaminofluorene	181	180	223
Dibenz(a,j)acridine	279	280	
7,12-Dimethylbenz(a)anthracene	256	241	120
3-Methylcholanthrene	268	252	253

TABLE 6: Method 8270C LCS Control Compounds

LCS Compounds	Spiking Level, Conc. Added = 20 ug/L
1,2,4-Trichlorobenzene	20
Acenaphthene	20
2,4-Dinitrotoluene	20
Pyrene	20
N-Nitroso-di-n-propylamine	20
1,4-Dichlorobenzene	20
Pentachlorophenol	20
Phenol	20
2-Chlorophenol	20
4-Chloro-3-methylphenol	20
4-Nitrophenol	20

TABLE 7

Method 8270C All Analyte Spike Mix	
Acenaphthene	100
Acenaphthylene	100
Anthracene	100
Benzo(a)anthracene	100
Benzo(b)fluoranthene	100
Benzo(k)fluoranthene	100
Benzo(a)pyrene	100
Benzo(ghi)perylene	100
Benzyl butyl phthalate	100
Bis(2-chloroethyl)ether	100
Bis(2-chloroethoxy)methane	100
Bis(2-ethylhexyl)phthalate	100
Bis(2-chloroisopropyl)ether	100

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Method 8270C All Analyte Spike Mix	
4-Bromophenyl phenyl ether	100
2-Chloronaphthalene	100
4-Chlorophenyl phenyl ether	100
Chrysene	100
Dibenzo(a,h)anthracene	100
Di-n-butylphthalate	100
1,3-Dichlorobenzene	100
1,2-Dichlorobenzene	100
1,4-Dichlorobenzene	100
3,3'-Dichlorobenzidine	100
Diethyl phthalate	100
Dimethyl phthalate	100
2,4-Dinitrotoluene	100
2,6-Dinitrotoluene	100
Di-n-octylphthalate	100
Fluoranthene	100
Fluorene	100
Hexachlorobenzene	100
Hexachlorobutadiene	100
Hexachloroethane	100
Indeno(1,2,3-cd)pyrene	100
Isophorone	100
Naphthalene	100
N-Nitrosodi-n-propylamine	100
Phenanthrene	100
Pyrene	100
1,2,4-Trichlorobenzene	100
4-Chloro-3-methylphenol	100
2-Chlorophenol	100
2,4-Dichlorophenol	100
2,4-Dimethylphenol	100
2,4-Dinitrophenol	100
2-Methyl-4,6-dinitrophenol	100
2-Nitrophenol	100
4-Nitrophenol	100
Pentachlorophenol	100
Phenol	100
2,4,6-Trichlorophenol	100

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Method 8270C All Analyte Spike Mix	
Acetophenone	100
Atrazine	100
Caprolactam	100
Benzaldehyde	100
1,1′-Biphenyl	100
Benzoic Acid	100
1,4-Dioxane	100
Benzyl Alcohol	100
Carbazole	100
4-Chloroaniline	100
Dibenzofuran	100
Hexachlorocyclopentadiene	100
2-Methylnaphthalene	100
1-Methylnaphthalene	100
2-Methylphenol	100
4-Methylphenol	100
4-Nitroaniline	100
2-Nitroaniline	100
3-Nitroaniline	100
Pyridine	100
2,3,5,6-Tetrachlorophenol	100
2,4,5-Trichlorophenol	100
N-Nitrosodimethylamine	100
N-Nitrosodiphenylamine	100

TABLE 8: TCLP LCS Compounds	
LCS Compounds	**Spiking Level, mg/L in extract**
1,4-Dichlorobenzene	0.08
2,4-Dinitrotoluene	0.08
Hexachlorobenzene	0.08
Hexachlorobutadiene	0.08
Hexachloroethane	0.08
2-Methylphenol	0.08
3-Methylphenol	0.08

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4-Methylphenol	0.08
Nitrobenzene	0.08
Pentachlorophenol	0.08
Pyridine	0.08
2,4,5-Trichlorophenol	0.08
2,4,6-Trichlorophenol	0.08

Recovery limits for the LCS and for matrix spikes are generated historical data, and are maintained by the QA Dept.

TABLE 9: Method 8270C Surrogate Compounds

Surrogate Compounds	Spiking Level, Conc. Added = 20 ug/L / 30 ug/L
Nitrobenzene-d5	20
2-Fluorobiphenyl	20
Terphenyl-d14	20
Phenol-d5	30
2-Fluorophenol	30
2,4,6-Tribromophenol	30

Recovery limits for surrogates are generated from historical data,
and are maintained by the QA department.

TABLE 10: Calibration Ranges

Analyte	Calibration Range
Pyridine	0.25-12.5 ug/mL
N-nitrosodimethylamine	0.25-12.5 ug/mL
Aniline	0.25-12.5 ug/mL
Phenol	0.25-12.5 ug/mL
Bis(2-chloroethyl)ether	0.25-12.5 ug/mL
2-Chlorophenol	0.25-12.5 ug/mL
1,3-Dichlorobenzene	0.25-12.5 ug/mL
1,4-Dichlorobenzene	0.25-12.5 ug/mL
Benzyl alcohol	0.25-12.5 ug/mL
1,2-Dichlorobenzene	0.25-12.5 ug/mL

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Analyte	Calibration Range
2-Methylphenol	0.25-12.5 ug/mL
2,2'-oxybis(1-chloropropane)[1]	0.25-12.5 ug/mL
4-Methylphenol	0.25-12.5 ug/mL
N-Nitroso-di-n-propylamine	0.25-12.5 ug/mL
Hexachloroethane	0.25-12.5 ug/mL
Nitrobenzene	0.25-12.5 ug/mL
Isophorone	0.25-12.5 ug/mL
2-Nitrophenol	0.25-12.5 ug/mL
2,4-Dimethylphenol	0.25-12.5 ug/mL
Benzoic acid	0.25-12.5 ug/mL
Bis(2-chloroethoxy)methane	0.25-12.5 ug/mL
2,4-Dichlorophenol	0.25-12.5 ug/mL
1,2,4-Trichlorobenzene	0.25-12.5 ug/mL
Naphthalene	0.05-10 ug/mL
4-Chloroaniline	0.25-12.5 ug/mL
Hexachlorobutadiene	0.25-12.5 ug/mL
4-Chloro-3-methylphenol	0.25-12.5 ug/mL
2-Methylnaphthalene	0.05-10 ug/mL
Hexachlorocyclopentadiene	0.25-12.5 ug/mL
2,4,6-Trichlorophenol	0.25-12.5 ug/mL
2,4,5-Trichlorophenol	0.25-12.5 ug/mL
2-Chloronaphthalene	0.25-12.5 ug/mL
2-Nitroaniline	0.25-12.5 ug/mL
Dimethyl phthalate	0.25-12.5 ug/mL
Acenaphthylene	0.05-10 ug/mL
3-Nitroaniline	0.25-12.5 ug/mL
Acenaphthene	0.05-10 ug/mL
2,4-Dinitrophenol	0.25-12.5 ug/mL
4-Nitrophenol	0.25-12.5 ug/mL
Dibenzofuran	0.25-12.5 ug/mL
2,4-Dinitrotoluene	0.25-12.5 ug/mL
2,6-Dinitrotoluene	0.25-12.5 ug/mL
Diethylphthalate	0.25-12.5 ug/mL
4-Chlorophenyl phenyl ether	0.25-12.5 ug/mL
Fluorene	0.05-10 ug/mL
4-Nitroaniline	0.25-12.5 ug/mL
4,6-Dinitro-2-methylphenol	0.25-12.5 ug/mL
N-Nitrosodiphenylamine	0.25-12.5 ug/mL
Azobenzene[2]	0.25-12.5 ug/mL

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Analyte	Calibration Range
4-Bromophenyl phenyl ether	0.25-12.5 ug/mL
Hexachlorobenzene	0.25-12.5 ug/mL
Pentachlorophenol	0.25-12.5 ug/mL
Phenanthrene	0.05-10 ug/mL
Anthracene	0.05-10 ug/mL
Carbazole	0.05-10 ug/mL
Di-n-butyl phthalate	0.25-12.5 ug/mL
Fluoranthene	0.05-10 ug/mL
Benzidine	0.25-12.5 ug/mL
Pyrene	0.05-10 ug/mL
Butyl benzyl phthalate	0.25-12.5 ug/mL
3,3'-Dichlorobenzidine	0.25-12.5 ug/mL

Analyte	Calibration Range
Benzo(a)anthracene	0.05-10 ug/mL
Bis(2-ethylhexyl)phthalate	0.25-12.5 ug/mL
Chrysene	0.05-10 ug/mL
Di-n-octylphthalate	0.25-12.5 ug/mL
Benzo(b)fluoranthene	0.05-10 ug/mL
Benzo(k)fluoranthene	0.05-10 ug/mL
Benzo(a)pyrene	0.05-10 ug/mL
Indeno(1,2,3-cd)pyrene	0.05-10 ug/mL
Dibenz(a,h)anthracene	0.05-10 ug/mL
Benzo(g,h,i)perylene	0.05-10 ug/mL
Benzaldehyde	0.25-12.5 ug/mL
Caprolactam	0.25-12.5 ug/mL
1,1'-Biphenyl	0.25-12.5 ug/mL
Atrazine	0.25-12.5 ug/mL
2-Picoline	0.25-12.5 ug/mL
N-Nitrosomethylethylamine	0.25-12.5 ug/mL
Methyl methanesulfonate	0.25-12.5 ug/mL
N-Nitrosodiethylamine	0.25-12.5 ug/mL
Ethyl methanesulfonate	0.25-12.5 ug/mL
Pentachloroethane	0.25-12.5 ug/mL
Acetophenone	0.25-12.5 ug/mL
N-Nitrosopyrrolidine	0.25-12.5 ug/mL
N-Nitrosomorpholine	0.25-12.5 ug/mL
o-Toluidine	0.25-12.5 ug/mL

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Analyte	Calibration Range
3-Methylphenol	0.25-12.5 ug/mL
N-Nitrosopiperidine	0.25-12.5 ug/mL
o,o,o-Triethyl-Phosphorothioate	0.25-12.5 ug/mL
a,a-Dimethyl-phenethylamine	0.25-12.5 ug/mL
2,6-Dichlorophenol	0.25-12.5 ug/mL
Hexachloropropene	0.25-12.5 ug/mL
p-Phenylenediamine	0.25-12.5 ug/mL
n-Nitrosodi-n-butylamine	0.25-12.5 ug/mL
Safrole	0.25-12.5 ug/mL
1,2,4,5-Tetrachlorobenzene	0.25-12.5 ug/mL
Isosafrole 1 + 2	0.25-12.5 ug/mL
1,4-Dinitrobenzene	0.25-12.5 ug/mL
1,4-Naphthoquinone	0.25-12.5 ug/mL
1,3-Dinitrobenzene	0.25-12.5 ug/mL
Pentachlorobenzene	0.25-12.5 ug/mL
1-Naphthylamine	0.25-12.5 ug/mL
2-Naphthylamine	0.25-12.5 ug/mL
2,3,4,6-Tetrachlorophenol	0.25-12.5 ug/mL
5-Nitro-o-toluidine	0.25-12.5 ug/mL
Thionazin	0.25-12.5 ug/mL
1,3,5-Trinitrobenzene	0.25-12.5 ug/mL
Sulfotepp	0.25-12.5 ug/mL
Phorate	0.25-12.5 ug/mL
Phenacetin	0.25-12.5 ug/mL
Diallate 1 + 2	0.25-12.5 ug/mL
Dimethoate	0.25-12.5 ug/mL
4-Aminobiphenyl	0.25-12.5 ug/mL
Pentachloronitrobenzene	0.25-12.5 ug/mL
Pronamide	0.25-12.5 ug/mL
Disulfoton	0.25-12.5 ug/mL
2-secbutyl-4,6-dinitrophenol (Dinoseb)	0.25-12.5 ug/mL
Methyl parathion	0.25-12.5 ug/mL
4-Nitroquinoline-1-oxide	0.25-12.5 ug/mL
Parathion	0.25-12.5 ug/mL
Isodrin	0.25-12.5 ug/mL
Kepone	0.25-12.5 ug/mL
Famphur	0.25-12.5 ug/mL
Methapyrilene	0.25-12.5 ug/mL

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Analyte	Calibration Range
Aramite 1 and 2	0.25-12.5 ug/mL
p-(Dimethylamino)azobenzene	0.25-12.5 ug/mL
p-Chlorobenzilate	0.25-12.5 ug/mL
3,3'-Dimethylbenzidine	0.25-12.5 ug/mL
2-Acetylaminofluorene	0.25-12.5 ug/mL
Dibenz (a,j)acridine	0.25-12.5 ug/mL
7,12-Dimethylbenz(a)anthracene	0.25-12.5 ug/mL
3-Methylcholanthrene	0.25-12.5 ug/mL

[1] 2,2'oxybis(1-chloropropane) was formerly known as bis(2-chloroisopropyl)ether.

[2] Azobenzene is formed by decomposition of 1,2-diphenyhydrazine. If 1,2-diphenylhydrazine is requested, it will be analyzed as azobenzene.

Note: Nine calibrations standards are prepared varying in concentration from 0.05 ug/mL to 12.5 ug/mL. A minimum of 5 calibration concentrations will be used for initial calibration . The concentration range of each analyte is listed in the table.

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Table 11A: Method 8270D Semivolatile Internal Standards with Corresponding Analytes Assigned for Quantitation

SEMIVOLATILE INTERNAL STANDARDS WITH CORRESPONDING ANALYTES ASSIGNED FOR QUANTITATION

1,4-Dichlorobenzene-d_4	Naphthalene-d_8	Acenaphthene-d_{10}
Aniline	Acetophenone	Acenaphthene
Benzyl alcohol	Benzoic acid	Acenaphthylene
Bis(2-chloroethyl) ether	Bis(2-chloroethoxy)methane	1-Chloronaphthalene
Bis(2-chloroisopropyl) ether	4-Chloroaniline	2-Chloronaphthalene
2-Chlorophenol	4-Chloro-3-methylphenol	4-Chlorophenyl phenyl ether
1,3-Dichlorobenzene	2,4-Dichlorophenol	Dibenzofuran
1,4-Dichlorobenzene	2,6-Dichlorophenol	Diethyl phthalate
1,2-Dichlorobenzene	α,α-Dimethyl-	Dimethyl phthalate
Ethyl methanesulfonate	phenethylamine	2,4-Dinitrophenol
2-Fluorophenol (surr)	2,4-Dimethylphenol	2,4-Dinitrotoluene
Hexachloroethane	Hexachlorobutadiene	2,6-Dinitrotoluene
Methyl methanesulfonate	Isophorone	Fluorene
2-Methylphenol	2-Methylnaphthalene	2-Fluorobiphenyl (surr)
4-Methylphenol	Naphthalene	Hexachlorocyclopentadiene
N-Nitrosodimethylamine	Nitrobenzene	1-Naphthylamine
N-Nitroso-di-n-propylamine	Nitrobenzene-d_8 (surr)	2-Naphthylamine
Phenol	2-Nitrophenol	2-Nitroaniline
Phenol-d_6 (surr)	N-Nitrosodi-n-butylamine	3-Nitroaniline
2-Picoline	N-Nitrosopiperidine	4-Nitroaniline
	1,2,4-Trichlorobenzene	4-Nitrophenol
		Pentachlorobenzene
		1,2,4,5-Tetrachlorobenzene
		2,3,4,6-Tetrachlorophenol
		2,4,6-Tribromophenol (surr)
		2,4,6-Trichlorophenol
		2,4,5-Trichlorophenol

(surr) = surrogate

Phenanthrene-d_{10}	Chrysene-d_{12}	Perylene-d_{12}
4-Aminobiphenyl	Benzidine	Benzo(b)fluoranthene
Anthracene	Benzo(a)anthracene	Benzo(k)fluoranthene
4-Bromophenyl phenyl ether	Bis(2-ethylhexyl) phthalate	Benzo(g,h,i)perylene
Di-n-butyl phthalate	Butyl benzyl phthalate	Benzo(a)pyrene
4,6-Dinitro-2-methylphenol	Chrysene	Dibenz(a,j)acridine
Diphenylamine	3,3'-Dichlorobenzidine	Dibenz(a,h)anthracene
Fluoranthene	p-Dimethyl aminoazobenzene	7,12-Dimethylbenz(a) anthracene
Hexachlorobenzene	Pyrene	Di-n-octyl phthalate
N-Nitrosodiphenylamine	Terphenyl-d_{14} (surr)	Indeno(1,2,3-cd) pyrene
Pentachlorophenol		3-Methylcholanthrene
Pentachloronitrobenzene		
Phenacetin		
Phenanthrene		
Pronamide		

(surr) = surrogate

Table 11B: Method 8270C Semivolatile Internal Standards with Corresponding Analytes Assigned for Quantitation

1,4-Dichlorobenzene-d4	Naphthalene-d8	Acenaphthene-d10
Bis(2-chloroethyl)ether	Bis(2-chloroethoxy)methane	Acenaphthene
Bis(2-chloroisopropyl)ether	2,4-Dichlorophenol	Acenaphthylene
2-Chlorophenol	2,4-Dimethylphenol	2-Chloronaphthalene
1,2-Dichlorobenzene	Hexachlorobutadiene	4-Chlorophenyl phenyl ether
1,3-Dichlorobenzene	Isophorone	Diethyl phthalate
1,4-Dichlorobenzene	Nitrobenzene	Dimethyl phthalate
2-Fluorophenol (surrogate)	Nitrobenzene-d_8 (surrogate)	2,4-Dinitrophenol
Hexachloroethane	2-Nitrophenol	2,4-Dinitrotoluene
N-Nitroso-di-n-propylamine	1,2,4-Trichlorobenzene	2,6-Dinitrotoluene
Phenol		Fluorene
Phenol-d6 (surrogate)		2-Fluorobiphenyl (surrogate)
		4-Nitrophenol
		2,4,6-Tribromophenol (surrogate)
		2,4,6-Trichlorophenol

Table 11C: Semivolatile Internal Standards with Corresponding Analytes Assigned for Quantitation

Phenanthrene-d10	Chrysene-d12	Perylene-d12
Anthracene	Benzo(a)anthracene	Benzo(b)fluoranthene
4-Bromophenyl phenyl ether	Bis(2-ethylhexyl)phthalate	Benzo(k)fluoranthene
Di-n-butyl phthalate	Chrysene	Benzo(g,h,i)perylene
4,6-Dinitro-2-methylphenol	3,3'-Dichlorobenzidine	Benzo(a)pyrene
Fluoranthene	Pyrene	Dibenz(a,h)anthracene
Hexachlorobenzene	Terphenyl-dl4 (surrogate)	Di-n-octylphthalate
Pentachlorophenol		Indeno(1,2,3-cd)pyrene
Phenanthrene		3-Methylcholanthrene

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Table 12: Recommended Minimum Response Factor Criteria for Initial and Continuing Calibration Verification

Semivolatile Compounds	Minimum Response Factor (RF)
Benzaldehyde	0.010
Phenol	0.800
Bis(2-chloroethyl)ether	0.700
2-Chlorophenol	0.800
2-Methylphenol	0.700
2,2'-Oxybis-(1-chloropropane)	0.010
Actophenone	0.010
4-Methylphenol	0.600
N-Nitros-di-n-propylamine	0.500
Hexachloroethane	0.300
Nitrobenzene	0.200
Isophorone	0.400
2-Nitrophenol	0.100
2,4-Dimethylphenol	0.200
Bis(2-chloroethoxy)methane	0.300
2,4-Dichlorophenol	0.200
Naphthalene	0.700
4-Chloroanaline	0.010
Hexachlorobutadiene	0.010
Caprolactuam	0.010
4-Chloro-3-methylphenol	0.200
2-Methylnaphthalene	0.400
Hexachlorocyclopentadiene	0.050
2,4,6-Trichlorophenol	0.200
2,4,5-Trichlorophenol	0.200
1,1'-Biphenyl	0.010
2-Chloronaphthalene	0.800
2-Nitroanaline	0.010
Dimethyl phthalate	0.010
2,6-Dinitrotoluene	0.200
Acenaphthylene	0.900
3-Nitroaniline	0.010
Acenaphthene	0.900
2,4-Dinitrophenol	0.010
4-Nitrophenol	0.010
Dibenzofuran	0.800
2,4-Dinitrotoluene	0.200
Dithyl phthalate	0.010
1,2,4,5-Tetrachlorobenzene	0.010

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Semivolatile Compounds	Minimum Response Factor (RF)
4-Chlorophenyl-phenyl ether	0.400
Fluorene	0.900
4-Nitroaniline	0.010
4,6-Dinitro-2-methylphenol	0.010
4-Bromophenyl-phenyl ether	0.100
N-Nitrosodiphenylamine	0.010
Hexachlorobenzene	0.100
Atrazine	0.010
Pentachlorophenol	0.050
Phananthrene	0.700
Anthracene	0.700
Carbazole	0.010
Di-n-butyl phthalate	0.010
Fluoranthene	0.600
Pyrene	0.600
Butyl benzyl phthalate	0.010
3,3-Dichlorobenzidine	0.010
Benzo(a)anthracene	0.800
Chrysene	0.700
Bis-(2-ethylhexyl)phthalate	0.010
Di-n-octyl phthalate	0.010
Benzo(b)fluoranthene	0.700
Benzo(k)fluoranthene	0.700
Benzo(a)pyrene	0.700
Indeno(1,2,3-cd)pyrene	0.500
Dibenz(a,h)anthracene	0.400
Benzo(g,h,I)perylene	0.500
2,3,4,6-Tetrachlorophenol	0.010

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Title: ACID DIGESTION FOR AQUEOUS SAMPLES

[Methods: SW846 3005A, 3010A, and MCAWW 200 Series]

Approvals (Signature/Date):

Technology Specialist	Date	Health & Safety Coordinator	Date
Quality Assurance Manager	Date	Technical Director	Date
Laboratory Director	Date		

This SOP was previously identified as SOP No. NC-IP-011, Rev 0, dated 01/07/09

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1. SCOPE AND APPLICATION

1.1. This procedure describes the preparation of aqueous samples for the analysis of certain metals by Inductively Coupled Plasma Atomic Emission Spectroscopy (ICP), and Inductively Coupled Plasma-Mass Spectrometry (ICPMS) using the MCAWW 200 series methods (NPDES) and SW846 Methods 3005A, and 3010A.

1.2. The applicability of each of these preparation protocols to specific analytes is detailed in Tables I and II (Appendix A). Additional elements may be analyzed following digestion by these protocols provided that the method performance criteria specified in Section 13.0 of this SOP are met.

1.3. This SOP provides procedures applicable to the preparation of dissolved, suspended, total recoverable and total elements in ground water, aqueous samples, certain aqueous sludges, and leachates/extracts.

1.4. SW-846 Method 3005A / MCAWW Method 200.8 are used to prepare surface and groundwater samples for total recoverable and dissolved metals determination by ICP and ICPMS.

1.5. MCAWW Method 200.7 is used to prepare surface water, domestic and industrial waste samples for total, total recoverable, and dissolved metals determination by ICP.

1.6. SW-846 Method 3010A is used to prepare aqueous samples, mobility-procedure extracts, and wastes that contain suspended solids for total metals analysis by ICP.

2. SUMMARY OF METHOD

2.1. Method 3005A / Method 200.7 / Method 200.8 - Preparation for Total Recoverable or Dissolved Metals Analysis by ICP and ICPMS

2.1.1 A representative aliquot of sample is heated with nitric and hydrochloric acids and reduced to a low volume. The digestate is filtered (if necessary) and diluted to volume.

2.2. Method 3010A / Method 200.7 / - Preparation for Total Metals Analysis by ICP

2.2.1 A representative aliquot of sample is refluxed with nitric acid. After the digestate has been reduced to a low volume, it is refluxed with 1:1 hydrochloric acid, filtered (if necessary), and diluted to volume.

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3. DEFINITIONS

3.1. Refer to the glossary in the TestAmerica North Canton Quality Assurance Manual (QAM), current version.

4. INTERFERENCES

4.1. There are numerous routes by which samples may become contaminated. Potential sources of trace metals contamination include metallic or metal-containing labware (e.g., talc gloves which contain high levels of zinc), containers, impure reagents, dirty glassware, improper sample transfers, dirty work areas, atmospheric inputs such as dirt and dust, etc. Be aware of potential sources of contamination and take appropriate measures to minimize or avoid them. All glassware is cleaned per SOP NC-QA-014.

4.2. The entire work area, including the bench top and fume hood, must be thoroughly cleaned on a routine schedule in order to minimize the potential for environmental contamination. Refer to Appendix B for additional contamination control guidelines.

4.3. Boron from the glassware will migrate into the sample solution during and following sample processing. For critical low level determinations of boron, it is recommended quartz or plastic labware be used.

4.4. Physical interference effects may contribute to inaccuracies in the determinations of trace elements. Oils, solvents, and other matrices may not be digested using these methods if they are not soluble with acids.

4.5. Visual interferences or anomalies (such as dilution due to oily matrix) must be documented.

4.6. Allowing samples to boil or go dry during digestion may result in the loss of volatile metals. If this occurs the sample must be reprepared. Antimony is easily lost by volatilization from hydrochloric acid media.

4.7. Precipitation of silver chloride (AgCl) may occur when chloride ions and high concentrations of silver (i.e., greater than 1 mg/L) are present in the sample.

4.8. Specific analytical interferences are discussed in each of the determinative methods.

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5. SAFETY

5.1. Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual, the Facility Addendum to the Corporate EH&S Manual, and this document.

5.2. Samples that contain high concentrations of carbonates or organic material or samples that are at elevated pH can react violently when acids are added.

5.3. The following is a list of the materials used in this method, which have a serious or significant hazard rating. **NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the Reagents and Standards section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Hydrochloric Acid	Corrosive Poison	5 ppm-Ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Nitric Acid	Corrosive Oxidizer Poison	2 ppm-TWA 4 ppm-STEL	Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison. Inhalation of vapors can cause breathing difficulties and lead to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract. Can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

5.4. Eye protection that protects against splash, laboratory coat, and appropriate gloves must be worn while samples, standards, solvents, and reagents are being handled. Cut-resistant

gloves must be worn doing any other task that presents a strong possibility of getting cut. Disposable gloves that have been contaminated must be removed and discarded; other gloves must be cleaned immediately.

5.5. The acidification of samples containing reactive materials may result in the release of toxic gases, such as cyanides or sulfides. Acidification of samples must be done in a fume hood. The analyst should also be aware of the potential for a vigorous reaction.

5.6. Exposure to chemicals must be maintained **as low as reasonably achievable.** All samples with stickers that read "Caution/Use Hood!" **must** be opened in the hood. Contact the EH&S Coordinator if this is not possible. Solvent and waste containers must be kept closed unless transfers are being made.

5.7. All work must be stopped in the event of a known or potential compromise to the health and safety of a TestAmerica associate. The situation must be reported **immediately** to the EH&S Coordinator and the Laboratory Supervisor.

5.8. Always carry bulk concentrated acid bottles in appropriate impact proof containers.

5.9. Acid spills must be neutralized immediately, flushed with water, and cleaned up using appropriate spill kits.

5.10. Discard chipped or broken beakers to prevent injury. Chipped glassware may be fire-polished as an alternative to disposal.

6. EQUIPMENT AND SUPPLIES

6.1. Hot plate, digestion block, or other adjustable heating source capable of maintaining a temperature of 90-95°C

6.2. Calibrated thermometer that covers a temperature range of 0-200°C

6.3. Griffin beakers of assorted sizes or equivalent. Equivalent containers may be disposable digestion cups for digestion blocks which are certified by the manufacturer and calibrated by the laboratory.

6.4. Watch glasses, ribbed or equivalent

6.5. Whatman No. 41 filter paper or equivalent

6.6. Funnels or equivalent filtration apparatus

6.7. Graduated cylinder or equivalent capable of measuring 50 mL within 3% accuracy

6.8. Analytical balance capable of accurately weighing to the nearest 0.01 grams

6.9. Repeaters or suitable reagent dispensers

6.10. Calibrated automatic pipettes with corresponding pipette tips

6.11. Class A volumetric flasks

6.12. pH indicator strips (pH range 0 - 6)

6.13. Plastic digestate storage bottles, such as Corning Snap Seals (may be used if their accuracy is documented and is better than 2%)

7. REAGENTS AND STANDARDS

7.1. Reagent water must be produced by a Millipore DI system or equivalent. Reagent water must be free of the analytes of interest as demonstrated through the analysis of method blanks as defined in the determinative SOPs.

7.2. Laboratory Control Sample (LCS) and matrix spike (MS) solutions are purchased as custom solutions. All standards must be stored in FEP fluorocarbon or previously unused polyethylene or polypropylene bottles. Stock standard solutions must be replaced prior to the expiration date provided by the manufacturer. If no expiration date is provided, the stock solutions may be used for up to one year and must be replaced sooner if verification from an independent source indicates a problem. Additional information can be found in SOP NC-QA-017.

7.3. Working ICP LCS/MS spike solution: Prepare the ICP LCS/MS working spike solution from custom stock standards to the final concentration listed in Table III. The working spike must be prepared in a matrix of 5% HNO_3. This acid (5 mL of concentrated HNO_3 per 100 mL) must be added to the volumetric flask before the addition of the stock standard aliquot. The working ICP LCS solution must be made fresh every three months.

7.4. The ICPMS LCS/MS spike solution is provided directly by the vendor. No further standard preparation is necessary.

7.5. The TCLP MS working spike solution is provided directly by the vendor, no further standard preparation is necessary. Refer to Table V for final digestate spike concentrations.

7.6. The LCS and MS samples must contain all the elements designated for analysis in each batch of samples. If a non-routine element is required that is not contained in the custom solution, the individual facility must purchase a solution from the designated vendor that will cover the additional analyte(s) of interest and provide for a final spike concentration that is appropriate to the determinative method.

7.7. Aqueous laboratory control samples (LCS) and matrix spike samples are prepared as described in Sections 9.5 and 9.6. Refer to Tables III and IV (Appendix A) for details regarding the stock, working standard and final digestate spike concentrations for ICP and ICPMS LCS and matrix spike preparations.

7.8. Nitric acid (HNO_3), concentrated, trace metal grade or better

7.9. Hydrochloric acid (HCl), concentrated, trace metal grade or better

7.10. Hydrochloric acid, 1:1 - dilute concentrated HCl with an equal volume of reagent water.

Note: When preparing diluted acids, always add acid to water. If the water is added to the acid, a violent reaction may occur.

8. SAMPLE COLLECTION, PRESERVATION AND STORAGE

8.1. Sample holding time for metals included under the scope of this SOP is 180 days from the date of collection to the date of analysis.

8.2. Aqueous samples are preserved with nitric acid to a pH of <2 and must be stored in either plastic or glass. If boron is to be determined, plastic containers are preferred. Refrigeration is not required. Preservation must be verified prior to analysis.

8.3. For dissolved metals analysis, the samples must be filtered through a 0.45 um filter prior to preservation. Filtration must be done in the field. In the event that samples are not field filtered, filtration occurs in the laboratory prior to preparation.

9. QUALITY CONTROL

Table VI (Appendix A) provides a summary of quality control requirements including type, frequency, acceptance criteria and corrective action.

9.1. Preparation Batch: A group of up to 20 samples that are of the same matrix and are processed together using the same procedures and reagents. The preparation batch must contain a method blank, an LCS, and a matrix spike/matrix spike duplicate. In some cases, at client request, it may be appropriate to process a matrix spike and sample duplicate in

place of the MS/MSD. If clients specify specific samples for MS/MSD, the batch may contain multiple MS/MSD pairs. In cases where there is insufficient sample volume to perform an MS/MSD, an LCS/LCS duplicate is required.

9.2. Sample Count: Laboratory generated QC samples (method blanks, LCS, MS/MSD) are not included in the sample count for determining the size of a preparation batch.

9.3. Method Blank (MB): One method blank must be processed with each preparation batch. The method blank consists of reagent water containing all reagents specific to the method that is carried through the entire analytical procedure, including preparation and analysis. The method blank is used to identify any system and process interferences or contamination of the analytical system that may lead to the reporting of elevated analyte concentrations or false positive data.

9.3.1 Aqueous method blanks are prepared by taking 50 mL of reagent water through the appropriate procedure as described in Section 11.

9.3.2 TCLP method blanks are prepared by taking 50 mL of leachate fluid through the appropriate procedure as described in Section 11.

9.4. Laboratory Control Sample (LCS): One aqueous LCS must be processed with each preparation batch. The LCS must contain all analytes of interest and must be carried through the entire analytical procedure. The LCS is used to monitor the accuracy of the analytical process. Ongoing monitoring of the LCS results provides evidence that the laboratory is performing the method within acceptable accuracy and precision guidelines. Criteria for the acceptance of LCS results are contained within the individual analytical method SOPs. Refer to Sections 7.3 and 7.4 for instructions on preparation of the aqueous LCS spike solution.

9.4.1 The aqueous LCS is prepared by spiking a 50 mL aliquot of reagent water with 1.0 mL for ICP and 0.5 mL for ICPMS of the working LCS/MS spike solution (Sections 7.3 or 7.4). The LCS is then processed through the appropriate procedure as described in Section 11.

Note: TCLP LCS is prepared by taking 50 mL of leachate fluid through the appropriate procedure as described in Section 11.

9.5. Additional information on QC samples can be found in QA Policy QA-003.

9.6. Matrix Spike/Matrix Spike Duplicate (MS/MSD): One MS/MSD pair must be processed for each preparation batch. A matrix spike (MS) is a field sample to which known

concentrations of target analytes have been added. A matrix spike duplicate (MSD) is a second aliquot of the same sample (spiked identically as the MS) prepared and analyzed along with the sample and matrix spike. Some client-specific data quality objectives (DQOs) may require the use of sample duplicates in place of or in addition to MS/MSDs. The MS/MSD results are used to determine the effect of a matrix on the precision and accuracy of the analytical process. Samples identified as field blanks cannot be used for MS/MSD analysis.

9.6.1 The aqueous matrix spike sample is prepared by spiking a 50 mL aliquot of a sample with 1.0 mL for ICP and 0.5 mL for ICPMS of the working LCS/MS spike solution (Sections 7.3 or 7.4). The matrix spike sample is then processed as described in Section 11.

9.6.2 The TCLP matrix spike sample is prepared by spiking a 50 mL aliquot of a leachate with 0.5 mL of the working TCLP spike solution (Section 7.5). The matrix spike sample is then processed as described in Section 11.

NOTE: The TCLP matrix spike must be added prior to preservation of the leachate.

NOTE: If analytes outside of the RCRA list are requested, l mL of additional spiking solution(s) are added.

9.6.3 If insufficient sample is available to process a MS/MSD, then a second LCS must be processed.

9.7. Control Limits

9.7.1 Control limits are established by the laboratory as described in SOP NC-QA-018.

9.7.2 Laboratory control limits are internally generated and updated periodically unless method specified. Control limits are easily accessible via LIMS (QC Browser program).

9.8 Method Detection Limits (MDLs) and MDL Checks

9.8.1 MDLs and MDL Checks are established by the laboratory as described in SOPs NC-QA-021 and CA-Q-S-006.

9.8.2 MDLs are easily accessible via LIMS (QC Browser program)

9.9 Nonconformance and Corrective Action

9.9.1 Any deviations from QC procedures must be documented as a nonconformance with applicable cause and corrective action approved by the facility QA Manager. Procedural deviations are not allowed for Ohio VAP projects.

10. CALIBRATION AND STANDARDIZATION

10.1 The hotplate/hotblock temperature must be verified daily for each hotplate used, and must be recorded on a hotplate/hotblock temperature log.

11. PROCEDURE

11.1. One-time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, chemistry, sample size, or other parameters. Any variation in procedure must be completely documented using a Nonconformance Memo. The Nonconformance Memo must be filed in the project file. Procedural deviations are not allowed for Ohio VAP projects.

11.2. All digestion procedures must be carried out in a properly functioning hood.

11.3. All samples are to be checked out of Sample Control with an electronic chain of custody.

11.4. Proper sample identification is extremely important in any preparation procedure. Labeling of beakers and bottles must be done in a manner to ensure connection with the proper sample.

11.5. Samples are typically logged in as either waters or soils. Wastes such as organic liquids or sludges and tissues (animal/vegetable) are usually logged in with solid test codes. When initiating prep, examine the sample to see if the sample matches the matrix designation. If the sample is logged in as aqueous but it appears more like a waste (biphasic, sludge like, organic liquid, lots of sediment, etc.), contact the lab supervisor or project manager for further instructions. In some cases, it may be more appropriate to process these samples as solids.

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11.6. If possible, prepare all the samples of a project at the same time to minimize the QC required and streamline the flow of the project through the lab and Reporting group.

11.7. Guidelines are provided in the appendices on procedures to minimize contamination of samples and standards.

11.8. For DoD work, refer to SOP NC-QA-016 for specific details.

11.9. The following procedure must be followed for all aqueous sample preparations.

11.9.1 Mix sample by shaking the container.

11.9.2 Measure 50 mL of the sample into a calibrated digestion tube. (Beakers may be used for oil matrices.)

11.9.3 Measure two extra aliquots of sample selected for the MS/MSD analysis. Spike each aliquot with the appropriate spiking solutions (Sections 7.3 to 7.5 and 9.6).

11.9.4 Measure 50 mL of reagent water into a calibrated digestion tube for the method blank.

11.9.5 Measure 50 mL of reagent water into a calibrated digestion tube for the LCS and add the appropriate spiking solutions (Sections 7.3 to 7.5 and 9.6).

11.10. Method 3005A / Method 200.7 / Method 200.8 - Preparation for Total Recoverable or Dissolved Metals Analysis by ICP / ICPMS

11.10.1 To the sample container, add 1 mL of concentrated HNO_3 and 2.5 mL of concentrated HCl.

11.10.2 Cover with ribbed watch glass.

11.10.3 Heat at 90-95°C until volume is reduced to between 15 and 20 mL. For DoD samples, the final volume is reduced to between 5 and 10 mL.

NOTE: DO NOT ALLOW SAMPLE TO BOIL OR GO DRY. Doing so will result in the loss of analyte and the sample must be reprepared.

11.10.4 Cool the beaker in a fume hood.

11.10.5 Filter sample, if insoluble materials are present, through Whatman 41 filter paper into a plastic storage container, such as a Corning Snap Seal™

Note: If any samples in a preparation batch are filtered, the method blank and LCS associated with that batch must also be filtered.

11.10.6 Rinse container and filter paper with reagent water to ensure complete sample transfer.

11.10.7 Adjust the final volume to 50 mL with reagent water in the Snap Seal™ container if the digestate was filtered or in the hot block digestion tube if filtering was not necessary. The sample is now ready for analysis.

11.11. Method 3010A / Method 200.7 - Preparation for Total Metals Analysis by ICP Spectroscopy

11.11.1 To the sample container, add 3.0 mL of concentrated HNO_3.

11.11.2 Cover with ribbed watch glass.

11.11.3 Place container on hotblock 90-95°C, and evaporate to a volume of 15-20 mL, while ensuring that no portion of the bottom of the beaker is allowed to go dry. For DoD projects, the sample is evaporated to 5-10 mL.

NOTE: DO NOT ALLOW SAMPLE TO BOIL OR GO DRY. Doing so will result in the loss of analyte and the sample must be re-prepared.

11.11.4 Add 5 mL of 1:1 HCl.

11.11.5 Cover and reflux for an additional 15 minutes to dissolve precipitate or residue. Cool in a fume hood.

11.11.6 Filter sample, if insoluble materials are present, through Whatman 41 filter paper into a plastic storage container, such as a Corning Snap Seal™.

Note: If any samples in the QC batch are filtered, the method blank and LCS associated with that batch must also be filtered.

11.11.7 Rinse container and filter paper with reagent water to ensure complete sample transfer.

11.11.8 Adjust final volume to 50 mL with reagent water in the Snap Seal™ container if the digestate was filtered, or in the hot block digestion tube if filtering was not necessary. The sample is now ready for analysis.

11.12. Analytical Documentation

11.12.1 Record all analytical information in the analytical logbook/logsheet, including the analytical data from standards, blanks, LCSs, MS/MSDs, and any corrective actions or modifications to the method.

11.12.2 All standards and reagents are logged into a department standard logbook. All standards are assigned a unique number for identification. Logbooks are reviewed by the supervisor or designee.

11.12.3 Documentation such as all associated instrument printouts (final runs, screens, reruns, QC samples, etc.) and daily calibration data corresponding to all final runs is available for each data file.

11.12.4 Sample results and associated QC are entered into LIMs after final technical review.

12. DATA ANALYSIS AND CALCULATIONS

Not applicable

13. METHOD PERFORMANCE

13.1. Initial Demonstration

13.1.1. Each laboratory must make an initial demonstration of capability for each individual method. This requires analysis of four QC Check samples

13.1.2. Four aliquots of the QC check sample are analyzed using the same procedures used to analyze samples, including sample preparation.

13.1.3. Calculations and acceptance criteria for the QC check samples are given in the determinative SOPs.

13.2. Training Qualification

13.2.1 The Group/Team Leader has the responsibility to ensure this procedure is performed by an associate who has been properly trained in its use and has the required experience.

13.2.2 Method validation information (where applicable) in the form of laboratory demonstrations of capabilities is maintained for this method in the laboratory QA files.

14. POLLUTION PREVENTION

14.1. It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage, and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention".

15. WASTE MANAGEMENT

15.1. All waste must be disposed of in accordance with Federal, State and Local laws and regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees must abide by this method and the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention".

15.2. Waste Streams Produced by the Method

15.2.1 The following waste streams are produced when this method is carried out.

15.2.1.1 Acidic waste containing nitric acid generated by the extraction. This waste is disposed of in the designated container labeled "Acid Waste".

15.2.1.2 Contaminated disposable materials utilized for the analysis. This waste is disposed of in a designated container identified as "Solid Waste".

15.3. Laboratory personnel assigned to perform hazardous waste disposal procedures must have a working knowledge of the established procedures and practices TestAmerica. They must have training on the hazardous waste disposal practices upon initial assignment to these tasks followed by an annual refresher training.

16. REFERENCES

16.1. References

16.1.1 Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition, Final Update I, Revision 1, July 1992. Methods 3005A and 3010A

16.1.2 Methods for the Chemical Analysis of Water and Waste (MCAWW), 1983

16.1.3 TestAmerica North Canton Quality Assurance Manual (QAM), current version

16.1.4 TestAmerica Corporate Environmental Health and Safety Manual, CW-E-M-001, and TestAmerica North Canton Facility Addendum and Contingency Plan, current version

16.1.5 Corporate Quality Management Plan (CQMP), current version

16.1.6 Revision History

Historical File:		Revision 1.2: 03/20/00		Revision 1.6: 02/07/07
(formerly CORP-IP-0003NC)		Revision 1.3: 09/25/01		Revision 0: 01/07/09 (NC-IP-011)
		Revision 1.4: 02/19/03		
		Revision 1.5: 12/07/04		

16.2. Associated SOPs and Policies, current version

16.2.1 TestAmerica QC Program, QA-003

16.2.2 Glassware Washing, NC-QA-014

16.2.3 Statistical Evaluation of Data and Development of Control Charts, NC-QA-018

16.2.4 Method Detection Limits and Instrument Detection Limits, NC-QA-021 and CA-Q-S-006

16.2.5 Supplemental Practices for DoD Project Work, NC-QA-016

16.2.6 Standards and Reagents, NC-QA-017

16.2.7 Subsampling, NC-IP-001

16.2.8 Inductively Coupled Plasma-Atomic Emission Spectroscopy, Spectrometric Method for Trace Element Analyses, SW846 Methods 6010A, 6010B, and 200.7, NC-MT-012

17. MISCELLANEOUS (TABLES, APPENDICES, ETC.)

17.1. Modifications/Interpretations from reference methods

17.1.1 Modifications applicable to SW-846 reference methods

17.1.1.1 The referenced methods as well as Table 3-1 of SW-846 refer to the use of a 100 mL aliquot for digestion. This SOP requires the use of a 50 mL sample size to reduce waste generation. The use of reduced sample volumes are supported in EPA's document "Response to Public Comments Background Document, Promulgation of the Second Update to SW-846, Third Edition" dated November 3, 1994. This document stated "flexibility to alter digestion volumes is addressed and "allowed" by Table 3-1 and is also inherently allowed by specific digestion methods. Table 3-1 is only to be used as guidance when collecting samples. EMSL-Ci has also taken the stance that "reduction in sample size and appropriate corresponding reduction in sample volume is not considered a significant change in the methodology." Additionally, in written correspondence from the Office of Solid Waste, Oliver Fordham stated " As a "representative sample" can be assured, scaling causes no loss of precision and accuracy in the analysis."

17.1.2 Modifications Specific to Method 3010A

17.1.2.1 Section 11.11.3 of this SOP requires the sample be reduced to a volume of 15 - 20 mL. Section 7.2 of Method 3010A states the volume should be reduced to 3 mL, but also states that no portion of the bottom of the beaker should go dry. The SOP required volume is a closer approximation of the volume required to provide an adequate covering of the beaker so as to prevent the loss of critical analytes through volatilization.

17.1.3 Modifications Specific to MCAWW Methods

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17.1.3.1 It was determined by technical review that several of the MCAWW methods were equivalent to the SW-846 methods and therefore were combined under the scope of this SOP as described in Section 11.0. The nature of the differences were deemed insignificant in regards to the amount of acid added and the evaporative volume based on the flexibility allowed by the methods (i.e., add additional acid as required) and the subjective wording of the methods (i.e., evaporate to near dryness vs. an exact volume).

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APPENDIX A - TABLES

TABLE I. Approved Preparation Method Analytes - SW846

ELEMENT	Symbol	CAS Number	3005A	3010A
Aluminum	Al	7429-90-5	X	X
Antimony	Sb	7440-36-0	X	
Arsenic	As	7440-38-2	X	X
Barium	Ba	7440-39-3	X	X
Beryllium	Be	7440-41-7	X	X
Cadmium	Cd	7440-43-9	X	X
Calcium	Ca	7440-70-2	X	X
Chromium	Cr	7440-47-3	X	X
Cobalt	Co	7440-48-4	X	X
Copper	Cu	7440-50-8	X	X
Iron	Fe	7439-89-6	X	X
Lead	Pb	7439-92-1	X	X
Magnesium	Mg	7439-95-4	X	X
Manganese	Mn	7439-96-5	X	X
Molybdenum	Mo	7439-98-7	X	X
Nickel	Ni	7440-02-0	X	X
Potassium	K	7440-09-7	X	X
Selenium	Se	7782-49-2	X	X
Silver	Ag	7440-22-4	X	X
Sodium	Na	7440-23-5	X	X
Thallium	Tl	7440-28-0	X	X
Vanadium	V	7440-62-2	X	X
Zinc	Zn	7440-66-6	X	X

X - Designates that the preparation method is approved for an element.

Note: Additional elements may be analyzed following digestion by these protocols provided the method performance criteria specified in Section 13.0 of the SOP are met.

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TABLE II. Approved Preparation Method Analytes – NPDES

ELEMENT	Symbol	CAS Number	200.7 (9.4)	200.7 (9.3)
Aluminum	Al	7429-90-5	X	X
Antimony	Sb	7440-36-0	X	X
Arsenic	As	7440-38-2	X	X
Boron	B	7440-42-8	X	X
Barium	Ba	7440-39-3	X	X
Beryllium	Be	7440-41-7	X	X
Cadmium	Cd	7440-43-9	X	X
Calcium	Ca	7440-70-2	X	X
Chromium	Cr	7440-47-3	X	X
Cobalt	Co	7440-48-4	X	X
Copper	Cu	7440-50-8	X	X
Iron	Fe	7439-89-6	X	X
Lead	Pb	7439-92-1	X	X
Magnesium	Mg	7439-95-4	X	X
Manganese	Mn	7439-96-5	X	X
Molybdenum	Mo	7439-98-7	X	X
Nickel	Ni	7440-02-0	X	X
Potassium	K	7440-09-7	X	X
Selenium	Se	7782-49-2	X	X
Silicon	Si	7631-86-9	X	X
Silver	Ag	7440-22-4	X	X
Sodium	Na	7440-23-5	X	X
Thallium	Tl	7440-28-0	X	X
Vanadium	V	7440-62-2	X	X
Zinc	Zn	7440-66-6	X	X

X - Designates that the preparation method is approved for an element

Note: Additional elements may be analyzed following digestion by these protocols provided the method performance criteria specified in Section 13.0 of the SOP are met.

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TABLE III. ICP Matrix Spike and Aqueous Laboratory Control Sample Levels

ELEMENT	Working LCS/MS Standard (mg/L)	Aqueous LCS/ MS Level * (ug/l)
Aluminum	100	2000
Antimony	25	500
Arsenic	100	2000
Barium	100	2000
Beryllium	2.5	50
Cadmium	2.5	50
Calcium	2500	50000
Chromium	10	200
Cobalt	25	500
Copper	12.5	250
Iron	50	1000
Lead	50	500
Magnesium	2500	50000
Manganese	25	500
Molybdenum	50	1000
Nickel	25	500
Potassium	2500	50000
Selenium	100	2000
Silver	2.5	50
Sodium	2500	50000
Thallium	100	2000
Vanadium	25	500
Zinc	25	500
Boron	50	1000
Tin	100	2000
Titanium	50	1000

* Levels shown indicate the spike concentration in the final digestate of the aqueous LCS or matrix spike based on the addition of 1.0 mL working spike (7.3) to 50 mL of sample.

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TABLE IV. ICPMS Aqueous LCS and Matrix Spike Levels

ELEMENT	Working LCS/MS Standard (mg/L)	Aqueous LCS/MS Level* (ug/L)
Aluminum	1000	10000
Antimony	10	100
Arsenic	10	100
Barium	10	100
Beryllium	10	100
Cadmium	10	100
Calcium	1000	10000
Chromium	10	100
Cobalt	10	100
Copper	10	100
Iron	1000	10000
Lead	10	100
Magnesium	1000	10000
Manganese	10	100
Molybdenum	10	100
Nickel	10	100
Potassium	1000	10000
Selenium	10	100
Silver	10	100
Sodium	1000	10000
Strontium	10	100
Thallium	10	100
Vanadium	10	100
Zinc	10	100
Boron	10	100
Tin	10	100
Titanium	10	100

* Levels shown indicate the spike concentration in the final digestate of the aqueous LCS or matrix spike based on the addition of 0.5 mL working spike (Section 7.4) to 50 mL of sample.

TABLE V. TCLP Reporting Limits, Regulatory Limits and Matrix Spike Levels for ICP

ELEMENT	RL (ug/L)	Regulatory Limit (ug/L)	Spike Level (ug/L)*
Arsenic	500	5000	5000
Barium	10000	100000	50000
Cadmium	100	1000	1000
Chromium	500	5000	5000
Lead	500	5000	5000
Selenium	250	1000	1000
Silver	500	5000	1000

* Levels shown indicate the spike concentration in the final digestate of the aqueous LCS or matrix spike based on the addition of 0.5 mL working spike (Section 7.5) to 50 mL of sample.

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TABLE VI. Summary of Quality Control Requirements

QC PARAMETER	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
Method Blank	One per sample preparation batch of up to 20 samples.	Refer to determinative SOPs: - NC-MT-0002 - NC-MT-012	Redigest and re-analyze samples associated with the method blank.
Laboratory Control Sample (LCS)	One per sample preparation batch of up to 20 samples.	Refer to determinative SOPs: - NC-MT-0002 - NC-MT-012	Redigest and re-analyze all samples associated with the LCS.
Matrix Spike	One per sample preparation batch of up to 20 samples.	Refer to determinative SOPs: - NC-MT-0002 - NC-MT-012	Reprep not required unless preparation error suspected.
Matrix Spike Duplicate	See Matrix Spike	Refer to determinative SOPs: - NC-MT-0002 - NC-MT-012	See Corrective Action for Matrix Spike.

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APPENDIX B

CONTAMINATION CONTROL GUIDELINES

The following procedures are strongly recommended to prevent contamination:

All glassware must be washed with detergent and tap water and rinsed with 1:1 nitric acid followed by deionized water.

Proper laboratory housekeeping is essential in the reduction of contamination in the metals laboratory. All work areas must be kept scrupulously clean.

Powdered or Latex Gloves must not be used in the metals laboratory since the powder contains zinc, as well as other metallic analytes. Only vinyl or nitrile gloves must be used in the metals laboratory.

Glassware must be periodically checked for cracks and etches and discarded if found. Etched glassware can cause cross contamination of any metallic analytes.

The following are helpful hints in the identification of the source of contaminants:

Reagents or standards can contain contaminants or be contaminated with the improper use of a pipette.

Improper cleaning of glassware can cause contamination.

Separate glassware if an unusually high sample is analyzed and soak with sulfuric acid prior to routine cleaning.

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Title: ACID DIGESTION FOR SOLID SAMPLES

[Method: SW846 Method 3050B]

Approvals (Signature/Date):

Technology Specialist	08/09/10 Date	Health & Safety Coordinator	07/28/10 Date
Quality Assurance Manager	08/09/10 Date	Laboratory Director	08/08/10 Date
Technical Director	08/09/10 Date		

This SOP was previously identified as SOP No. NC-IP-010, Rev 1, dated 01/07/09

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1. SCOPE AND APPLICATION

1.1. This procedure describes the preparation of soil samples for the analysis of certain metals by Inductively Coupled Plasma Atomic Emission Spectroscopy (ICP) and Inductively Coupled Plasma-Mass Spectrometry (ICPMS) as specified in SW846 Method 3050B.

1.2. Samples prepared by the protocols detailed in this SOP may be analyzed by ICP or ICPMS for the elements listed in Table I (Appendix A). Other elements and matrices may be analyzed following digestion by these protocols provided that the method performance criteria specified in Section 13.0 of this SOP are met.

1.3. This method is not a total digestion, but will dissolve almost all metals that could become "environmentally available". By design, metals bound in silicate structures are not dissolved by this procedure, as they are not usually mobile in the environment. This SOP can be applied to metals in solids, sludges, wastes, sediments, biological samples, and wipes.

1.4. This document accurately reflects current laboratory Standard Operating Procedures (SOP) as of the date above. All facility SOPs are maintained and updated as necessary.

2. SUMMARY OF METHOD

2.1. A representative 1 gram (wet weight) portion of sample is digested in nitric acid and hydrogen peroxide. The digestate is refluxed with hydrochloric acid for ICP and ICPMS analysis. The digestates are then filtered and diluted to 100 mL.

3. DEFINITIONS

3.1. Refer to the glossary in the TestAmerica North Canton Quality Assurance Manual (QAM), latest version.

4. INTERFERENCES

4.1. There are numerous routes by which samples may become contaminated. Potential sources of trace metals contamination include metallic or metal-containing labware (e.g., talc gloves which contain high levels of zinc), containers, impure reagents, dirty glassware, improper sample transfers, dirty work areas, atmospheric inputs such as dirt and dust, etc. Be aware of potential sources of contamination, and take appropriate measures to minimize or avoid them. All glassware is cleaned per SOP NC-QA-014.

4.2. The entire work area, including the bench top and fume hood, must be thoroughly cleaned on a routine schedule in order to minimize the potential for environmental contamination.

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4.3. Boron from the glassware may leach into the sample solution during, and following, sample processing. For critical low-level determinations of boron, only quartz and/or plastic labware are recommended.

4.4. Visual interferences or anomalies, such as foaming, emulsions, precipitates, etc., must be documented.

4.5. Allowing samples to boil or go dry during digestion may result in the loss of volatile metals. If this occurs, the sample must be reprepared. Antimony is easily lost by volatilization from hydrochloric media.

4.6. Specific analytical interferences are discussed in each of the determinative methods.

5. SAFETY

5.1. Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual, the Facility Addendum to the Corporate EH&S Manual, and this document.

5.2. Samples that contain high concentrations of carbonates or organic material or samples that are at elevated pH can react violently when acids are added.

5.3. The following is a list of the materials used in this method, which have a serious or significant hazard rating. **NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the Reagents and Standards section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Hydrochloric Acid	Corrosive Poison	5 ppm-Ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Hydrogen Peroxide	Oxidizer Corrosive	1 ppm-TWA	Vapors are corrosive and irritating to the respiratory tract. Vapors are very corrosive and irritating to the eyes and skin.

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Nitric Acid	Corrosive Oxidizer Poison	2 ppm-TWA 4 ppm-STEL	Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison. Inhalation of vapors can cause breathing difficulties and lead to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract. Can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

5.4 Eye protection that protects against splash, laboratory coat, and appropriate gloves must be worn while samples, standards, solvents, and reagents are being handled. Cut-resistant gloves must be worn doing any other task that presents a strong possibility of getting cut. Disposable gloves that have been contaminated must be removed and discarded; other gloves must be cleaned immediately.

5.5 The acidification of samples containing reactive materials may result in the release of toxic gases, such as cyanides or sulfides. Acidification of samples must be done in a fume hood. The analyst must also be aware of the potential for a vigorous reaction.

5.6 Exposure to chemicals must be maintained **as low as reasonably achievable.** All samples with stickers that read "Caution/Use Hood!" **must** be opened in the hood. Contact the EH&S Coordinator if this is not possible. Solvent and waste containers must be kept closed unless transfers are being made.

5.7 All work must be stopped in the event of a known or potential compromise to the health and safety of a TestAmerica North Canton associate. The situation must be reported **immediately** to the EH&S Coordinator and the Laboratory Supervisor.

5.8 Always carry bulk concentrated acid bottles in appropriate impact proof containers.

5.9 Acid/peroxide spills must be neutralized immediately, flushed with water and cleaned up using appropriate spill kits.

5.10 Discard chipped or broken beakers to prevent injury. Chipped glassware may be fire polished as an alternative to disposal.

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6. EQUIPMENT AND SUPPLIES

6.1. Hot plate, digestion block, steam bath, or other heating source capable of maintaining a temperature of 91-99°C

6.2. Calibrated thermometer that covers a temperature range of 0-200°C

6.3. Vapor recovery device (Watch glasses, ribbed or other device)

6.4. Whatman No. 41 filter paper or equivalent

6.5. Funnels or equivalent filtration apparatus

6.6. Analytical balance capable of accurately weighing to the nearest 0.01 grams

6.7. Repeaters or suitable reagent dispensers

6.8. Calibrated automatic pipettes with corresponding pipette tips: 100uL, 500uL, 1mL-5mL

6.9. Class A volumetric flasks

6.10. Plastic digestate storage bottles, such as Corning Snap Seals™ (may be used if their accuracy is documented and is better than 2%)

6.11. Boiling Stones: Ultra Pure PTFE or equivalent

7. REAGENTS AND STANDARDS

7.1. Reagent water must be produced by a Millipore DI system or equivalent. Reagent water must be free of the analytes of interest as demonstrated through the analysis of method blanks as defined in the determinative SOPs.

7.2. Laboratory Control Sample (LCS) and matrix spike (MS) solutions are purchased as custom solutions. All standards must be stored in FEP fluorocarbon or previously unused polyethylene or polypropylene bottles. Stock standard solutions must be replaced prior to the expiration date provided by the manufacturer. If no expiration date is provided, the stock solutions may be used for up to one year and must be replaced sooner if verification from an independent source indicates a problem. Additional information can be found in SOP NC-QA-017.

7.3. Working ICP LCS/MS spike solution. Prepare the ICP LCS/MS working spike solutions from custom stock standards to the final concentration listed in Table II. The working spike must be prepared in a matrix of 5% HNO_3. This acid (5 mL of concentrated HNO_3 per 100 mL) must be added to the volumetric flask before the addition of the stock standard aliquot. The working ICP LCS solution must be made fresh every three months. Refer to the Standards

Logbook for details on standard preparation.

7.4. ICPMS LCS/MS spike solution. Laboratory Control Sample (LCS) and matrix spike (MS) solutions are custom made so the final concentrations after spiking equals the concentrations listed in Table III.

7.5. The LCS and MS samples must contain all the elements designated for analysis in each batch of samples. If a non-routine element is required that is not contained in the custom solution, the individual facility must purchase a solution from the designated vendor that will cover the additional analyte(s) of interest and provide for a final spike concentration that is appropriate to the determinative method.

7.6. Nitric acid (HNO_3), concentrated, trace metal grade or better

7.7. Nitric acid, 1:1 - dilute concentrated HNO_3 with an equal volume of reagent water

Note: When preparing diluted acids, always add acid to water. If the water is added to the acid a violent reaction may occur.

7.8. Hydrochloric acid (HCl), concentrated, trace metal grade or better

7.9. 30% Hydrogen peroxide (H_2O_2), Ultrapure grade

8. SAMPLE COLLECTION, PRESERVATION, AND STORAGE

8.1. Sample holding time for metals included under the scope of this SOP is 180 days from the date of sample collection to the date of analysis.

8.2. Soil samples do not require preservation.

9. QUALITY CONTROL

Table IV (Appendix A) provides a summary of quality control requirements including type, frequency, acceptance criteria, and corrective action.

9.1. Initial Demonstration of Capability

Prior to analysis of any analyte using Method 3050B the following requirements must be met.

9.1.1. Method Detection Limit (MDL) - An MDL must be determined for each analyte/matrix prior to the analysis of any samples. The result of the MDL determination must be below the TestAmerica North Canton reporting limit. Criteria for DoD work is noted in SOP NC-QA-016.

9.1.2. Initial Demonstration Study - This requires the analysis of four QC check samples.

The QC check sample is a well-characterized laboratory generated sample used to monitor method performance, which must contain all the analytes of interest. The results of the initial demonstration study must be acceptable before analysis of samples may begin. The results of the initial demonstration study may be used to extend a method for the analysis of other elements provided all acceptance criteria are met.

9.1.2.1. Four aliquots of the check sample (LCS) are prepared and analyzed using the procedures detailed in this SOP and the determinative SOPs.

9.1.2.2. Calculations and acceptance criteria for QC check samples are given in the determinative SOP NC-MT-012.

9.2. Preparation Batch - A group of up to 20 samples that are of the same matrix and are processed together using the same procedures and reagents. The preparation batch must contain a method blank, an LCS, and a matrix spike/matrix spike duplicate. In some cases, at client request, it may be appropriate to process a matrix spike and sample duplicate in place of the MS/MSD. If clients specify specific samples for MS/MSD, the batch may contain multiple MS/MSD pairs.

9.3. Sample Count - Laboratory generated QC samples (method blanks, LCS, MS/MSD) are not counted towards the maximum 20 samples in a batch. Field QC samples are included in the batch count.

9.4. Method Blank (MB) - One method blank must be processed with each preparation batch. The method blank consists of reagent water containing all reagents specific to the method that is carried through the entire analytical procedure, including preparation and analysis. The method blank is used to identify any system and process interferences or contamination of the analytical system that may lead to the reporting of elevated analyte concentrations or false positive data. Criteria for the acceptance of blanks are contained within the individual analytical method SOPs. If the method blank does not meet the criteria contained within the analytical method SOPs, the blank and all associated samples in the batch must be redigested.

9.4.1. The MB is prepared by weighing a 1g aliquot of boiling chips. The MB is then processed as described in Section 11.9.

9.5. Laboratory Control Sample (LCS) - One aqueous LCS must be processed with each preparation batch. The LCS must contain all analytes of interest and must be carried through the entire analytical procedure. The LCS is used to monitor the accuracy of the analytical process. Ongoing monitoring of the LCS results provides evidence that the laboratory is performing the method within acceptable accuracy and precision guidelines. Criteria for the acceptance of LCS results are contained within the individual analytical method SOPs. Corrective action when LCS results fail to meet control limits will be repreparation and reanalysis of the batch. Table II provide the details regarding the stock, working standards, and final spike concentrations for ICP and ICPMS. Refer to Section 7 for instructions on preparation of the aqueous LCS.

9.5.1. The ICP LCS is prepared by spiking a 1g aliquot of boiling chips with 2 mL of the working LCS/MS spike solution (Section 7.4). The ICPMS LCS is prepared by spiking a 1g aliquot of boiling chips with 1 mL of the LCS/MS solution (Section 7.4). The LCS is then processed as described in Section 11.9.

9.6. Additional information on QC samples can be found in QA Policy QA-003. Ohio VAP projects must reference this SOP instead of policy QA-003 for information on QC samples.

9.7. Matrix Spike/Matrix Spike Duplicate (MS/MSD) - One MS/MSD pair must be processed for each preparation batch. A matrix spike (MS) is a field sample to which known concentrations of target analytes have been added. A matrix spike duplicate (MSD) is a second aliquot of the same sample (spiked identically as the MS) prepared and analyzed along with the sample and matrix spike. Some client specific data quality objectives (DQOs) may require the use of sample duplicates in place of or in addition to MS/MSDs. The MS/MSD results are used to determine the effect of a matrix on the precision and accuracy of the analytical process. Samples identified as field blanks cannot be used for MS/MSD analysis. If any analyte recovery or RPD falls outside the acceptance range, the recovery of that analyte must be in control for the LCS. If the recovery of the LCS is outside limits, corrective action must be taken. Corrective action will include repreparation and reanalysis of the batch. Corrective action when MS results fail to meet control limits does not include repreparation of samples unless the results indicate that a spiking error may have occurred. Table II provides the details regarding the stock, working standards and final matrix spike concentrations for ICP and ICPMS. Refer to Section 7for instructions on preparation of the working matrix spike solutions.

9.7.1. The ICP matrix spike is prepared by spiking a 1g aliquot of sample with 2 mL of the working LCS/MS spike solution (Section 7). The ICPMS matrix spike is prepared by spiking a 1g aliquot of sample with 1 mL of the LCS/MS solution (Section 7.4). The matrix spike is then processed as described in Section 11.9.

10. CALIBRATION AND STANDARDIZATION

10.1. Hotblock temperature must be verified daily for each unit used, and must be recorded in a hotblock temperature log.

11. PROCEDURE

11.1. One-time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, chemistry, sample size, or other parameters. Any variation in procedure must be completely documented using a Nonconformance Memo. The Nonconformance Memo must be filed in the project file.

11.2. Any unauthorized deviations from this procedure must also be documented as a nonconformance with a cause and corrective action described. Deviations are not allowed for Ohio VAP projects.

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11.3. The heating procedures are carried out in a properly functioning hood.

11.4. All samples are to be checked out of Sample Control with an electronic Chain-of-Custody.

11.5. Proper sample identification is extremely important in any preparation procedure. Labeling of beakers and bottles must be done in a manner to ensure connection with the proper sample. An automatic label printing program is used to reduce transcription errors (QuantIMS option).

11.6. Samples are typically logged in as either waters or soils. Wastes, such as organic liquids or sludges and tissues (animal/vegetable), are usually logged in with solid test codes. When initiating prep, examine the sample to see if the sample matches the matrix designation.

11.7. If possible, prepare all the samples of a project at the same time to minimize the QC required and streamline the flow of the project through the lab and Reporting Group.

11.8. Guidelines are provided in the appendices on procedures to minimize contamination of samples and standards. Refer to Appendix B for details.

11.9. Preparation of Soils, Sediments, and Sludges for Analysis by ICP and ICPMS

11.9.1. Mix sample thoroughly by stirring with a clean plastic or wooden spoon or spatula.

11.9.2. For each digestion procedure required (i.e., ICP or ICPMS), weigh a 1g portion of solid and record the exact weight to the nearest 0.01g. A 2g sample size may also be used if needed to meet the reporting limits.

Note: Wipe samples are not weighed. The entire wipe is used.

11.9.3. Measure additional aliquots of the designated samples for the MS and MSD analyses.

11.9.4. Add 10 mL of 1:1 HNO_3 and mix the sample.

11.9.5. Heat sample to $95^{o} \pm 4°$ C and reflux for 10 minutes without boiling, using a vapor recovery device.

Note: DO NOT ALLOW SAMPLE TO BOIL OR GO DRY during any part of the digestion. Doing so will result in the loss of analyte and the sample must be reprepared.

11.9.6 Add 5 mL of concentrated HNO_3.

11.9.7 Reflux at $95^{o} \pm 4°$ C for 30 minutes. (Add reagent water, as needed, to ensure that the volume of solution is not reduced to less than 5 mL.)

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11.9.8 Add approximately 2 mL of reagent water and 1 mL of 30 % H_2O_2. Care must be taken to ensure that losses do not occur due to excessively vigorous effervescence.

11.9.9 Continue adding 30% H_2O_2 in 1 mL aliquots until effervescence is minimal or sample appearance is unchanged. Make sure effervescence subsides before each addition of H_2O_2.

Note: Do not add more than a total of 10 mL of 30 % H_2O_2.

11.9.10 Continue heating at $95^o \pm 4°$ C until the volume is reduced by cooking two hours or to approximately 5-10 mL.

11.9.11 Add 10 mL of concentrated HCL and reflux for an additional 15 minutes without boiling.

11.9.12 Allow the sample to cool.

11.9.13 Filter sample through Whatman 41 filter paper or equivalent, that has been rinsed with deionized water, into a measuring bottle (for example, Corning Snap Seals™). These may be used if their accuracy is documented and is better than $\pm$ 2%. Rinse sample container and filter paper with reagent water to ensure complete sample transfer.

11.9.14 Dilute sample to 100 mL with reagent water into a 120 mL graduated Snap Seal. The sample is now ready for analysis.

11.10. Analytical Documentation

11.10.1 Record all analytical information in the analytical logbook/logsheet, including the analytical data from standards, blanks, LCSs, MS/MSDs, and any corrective actions or modifications to the method.

11.10.2 All standards and reagents are logged into a department standard logbook. All standards are assigned a unique number for identification. Logbooks are reviewed by the supervisor or designee.

12. DATA ANALYSIS AND CALCULATIONS

Not applicable

13. METHOD PERFORMANCE

13.1. Method performance is determined by the analysis of matrix spike and matrix spike duplicate samples as well as method blanks and laboratory control samples. Acceptance criteria are given in the determinative SOPs.

13.2. The initial demonstration study as detailed in Section 9.1.2 must be acceptable before the analysis of field samples under this SOP may begin. The results of the initial demonstration study may be used to extend a method for the analysis of other elements provided all acceptance criteria are met.

13.3. Training Qualification

The Group/Team Leader has the responsibility to ensure this procedure is performed by an associate who has been properly trained in its use and has the required experience.

14. POLLUTION PREVENTION

14.1. It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recyling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention".

15. WASTE MANAGEMENT

15.1. All waste will be disposed of in accordance with Federal, State and Local laws and regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention".

15.2. Waste Streams Produced by the Method

15.2.1. The following waste streams are produced when this method is carried out.

15.2.1.1. Acidic waste containing nitric acid generated by the extraction. This waste is disposed of in a designated container labeled "Acid Waste".

15.2.1.2. Contaminated disposable materials utilized for the analysis. This waste is disposed of in a designated container labeled "Solid Waste".

16. REFERENCES

16.1. References

16.1.1. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition, Final Update III, December 1996, Method 3050B

16.1.2. TestAmerica North Canton Quality Assurance Manual (QAM), current version

16.1.3. TestAmerica Corporate Environmental Health and Safety Manual, CW-E-M-001, and TestAmerica North Canton Facility Addendum and Contingency Plan, current version

16.1.4. Corporate Quality Management Plan (CQMP), current version

16.1.5. Revision History

Historical File:		Revision 2.1: 02/11/00		Revision 0: 07/18/08 (NC-IP-010)
(formerly CORP-IP0002NC)		Revision 2.2: 09/25/01		Revision 1: 01/07/09
		Revision 2.3: 01/18/02		
		Revision 2.4: 02/19/03		
		Revision 2.5: 12/02/04		
		Revision 2.6: 07/29/07		

16.2. Associated SOPs and Policies, current version

16.2.1. Inductively Coupled Plasma-Atomic Emission Spectroscopy, Spectrometric Method for Trace Element Analysis of Water and Wastes, Methods 6010B and 200.7, NC-MT-012

16.2.2. Inductively Coupled Plasma-Mass Spectrometry, EPA Methods 6020 and 200.8, NC-MT-002

16.2.3. TestAmerica North Canton Quality Control Program, QA-003

16.2.4. Glassware Washing, NC-QA-014

16.2.5. Statistical Evaluation of Data and Development of Control Charts, NC-QA-018

16.2.6. Method Detection Limits and Instrument Detection Limits, NC-QA-021 and CA-Q-S-006

16.2.7. Supplemental Practices for DOD Project Work, NC-QA-016

16.2.8. Standards and Reagents, NC-QA-017

16.2.9. Subsampling, NC-IP-001

17. MISCELLANEOUS (TABLES, APPENDICES, ETC.)

17.1. Method Deviations

17.1.1. The laboratory uses the same preparation procedure for Method 6010B and 6020. Hydrochloric acid can be used for Method 6020 due to the collision cell technology on newer instruments. Due to the potential chloride interferences, and possible inability to analyze for arsenic and tin, the laboratory must follow the instrument manufacturer guidelines pertaining to the use of HCL and ICP/MS analyses.

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APPENDIX A: TABLES

TABLE I

Method 3050A Approved Analyte List

Element	Symbol	CAS Number
Aluminum	Al	7429-90-5
Antimony	Sb	7440-36-0
Arsenic	As	7440-38-2
Barium	Ba	7440-39-3
Beryllium	Be	7440-41-7
Cadmium	Cd	7440-43-9
Calcium	Ca	7440-70-2
Chromium	Cr	7440-47-3
Cobalt	Co	7440-48-4
Copper	Cu	7440-50-8
Iron	Fe	7439-89-6
Lead	Pb	7439-92-1
Magnesium	Mg	7439-95-4
Manganese	Mn	7439-96-5
Molybdenum	Mo	7439-98-7
Nickel	Ni	7440-02-0
Potassium	K	7440-09-7
Selenium	Se	7782-49-2
Silver	Ag	7440-22-4
Sodium	Na	7440-23-5
Thallium	Tl	7440-28-0
Vanadium	V	7440-62-2
Zinc	Zn	7440-66-6

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TABLE II

ICP Soil Matrix Spike and LCS Levels

Element	Working LCS/MS Standard (mg/L)	Soil MS/LCS Level * (mg/kg)
Aluminum	100	200
Antimony	25	50
Arsenic	100	200
Barium	100	200
Beryllium	2.5	5
Cadmium	2.5	5
Calcium	2500	5000
Chromium	10	20
Cobalt	25	50
Copper	12.5	25
Iron	50	100
Lead	25	50
Magnesium	2500	5000
Manganese	25	50
Molybdenum	50	100
Nickel	25	50
Potassium	2500	5000
Selenium	100	200
Silver	2.5	5
Sodium	2500	5000
Thallium	100	200
Vanadium	25	50
Zinc	25	50
Boron	50	100
Tin	100	200
Titanium	50	100

* Final soil spike concentration based on the addition of 2.0 mL working spike (Section 7.3) to 1.0 g of sample/100 mL final volume (assumes 100% solids).

TABLE III

ICPMS Soil Matrix Spike and LCS Levels

Element	Working LCS/MS Standard (mg/kg)	Soil MS/LCS Level * (mg/kg)
Aluminum	1000	1000
Antimony	10	10
Arsenic	10	10
Barium	10	10
Beryllium	10	10
Cadmium	10	10
Calcium	1000	1000
Chromium	10	10
Cobalt	10	10
Copper	10	10
Iron	1000	1000
Lead	10	10
Magnesium	1000	1000
Manganese	10	10
Molybdenum	10	10
Nickel	10	10
Potassium	1000	1000
Selenium	10	10
Silver	10	10
Sodium	1000	1000
Strontium	10	10
Thallium	10	10
Vanadium	10	10
Zinc	10	10
Boron	10	10
Tin	10	10
Titanium	10	10

* Final soil spike concentration based on the addition of 1.0 mL working spike (Section 7.4) to 1.0 g of sample/100 mL final volume (assumes 100% solids).

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TABLE IV

Summary of Quality Control Requirements

QC PARAMETER	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
Method Blank	One per sample preparation batch of up to 20 samples	Refer to determinative SOPs: NC-MT-002 NC-MT-012	Redigest and re-analyze samples
Laboratory Control Sample (LCS)	One per sample preparation batch of up to 20 samples	Refer to determinative SOPs: NC-MT-002 NC-MT-012	Redigest and re-analyze all samples associated with the LCS
Matrix Spike	One per sample preparation batch of up to 20 samples	Refer to determinative SOPs: NC-MT-002 NC-MT-012	Reprep not required unless preparation error suspected
Matrix Spike Duplicate	See Matrix Spike	Refer to determinative SOPs: NC-MT-002 NC-MT-012	See Corrective Action for Matrix Spike

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APPENDIX B. CONTAMINATION CONTROL GUIDELINES

The following procedures are strongly recommended to prevent contamination:

All glassware must be washed with detergent and tap water and rinsed with 1:1 nitric acid followed by deionized water.

Proper laboratory housekeeping is essential in the reduction of contamination in the Metals Lab. All work areas must be kept scrupulously clean.

Powdered gloves must not be used in the Metals Lab since the powder contains zinc, as well as other metallic analytes.

Glassware must be periodically checked for cracks and etches and discarded if found. Etched glassware can cause cross contamination of any metallic analytes.

The following are helpful hints in the identification of the source of contaminants:

Reagents or standards can contain contaminants or be contaminated with the improper use of a pipette.

Improper cleaning of glassware can cause contamination.

Separate glassware if an unusually high sample is analyzed and soak with sulfuric acid prior to routine cleaning.

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Title: CYANIDE PREPARATION METHOD

[Method: EPA Methods 335.1, 335.2, 335.2(CLP-M), 335.4, Standard Method 4500-CN-I, 4500-CN-E, 4500-CN-G, and SW846 Method 9012A]

Approvals (Signature/Date):

Technology Specialist	05/26/10 Date	Health & Safety Coordinator	05/25/10 Date
Quality Assurance Manager	05/25/10 Date	Laboratory Director	05/25/10 Date

This SOP was previously identified as SOP No. NC-WC-032, Rev 8.6, dated 12/30/08

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1. SCOPE AND APPLICATION

1.1. This method is applicable to the determination of Total, Amenable, and Weak Acid Dissociable in solids, liquids, and waters. It is based on SW 846 Method 9012A, EPA 335.1, 335.2, 335.2 (CLP-M) Ohio VAP only, 335.4, and Standard Methods 4500-CN-G and 4500-CN-I. The working linear range is 0.005 - 0.2 mg/L for waters and 0.25 to 10 mg/kg for solids.

1.2. The associated method codes are SW846 Method 9012A (QP), EPA 335.1 (CF) and 335.2 (CG), 335.2(CLP-M) (14) Ohio VAP only, EPA 335.4 (O2), Standard Method 4500-CN-E (A4), Weak Acid Dissociable 4500CN-I (HF), Amenable Cyanide 9012A (N4), and Standard Method 4500-CN-G (80).

1.3. This document accurately reflects current laboratory Standard Operating Procedures (SOP) as of the date above. All facility SOPs are maintained and updated as necessary by the laboratory.

2. SUMMARY OF METHOD

2.1. The Cyanide, as HCN, is released by distilling/refluxing the sample with strong acid and is trapped in a sodium hydroxide solution.

3. DEFINITIONS

3.1. Refer to the glossary in the TestAmerica North Canton Quality Assurance Manual (QAM), current version.

4. INTERFERENCES

4.1. Method interferences may be caused by contaminants in solvents, reagents, glassware, and other processing apparatus that lead to discrete artifacts. All of these materials must be routinely demonstrated to be free from interferences under conditions of the analysis by running laboratory method blanks as described in the Quality Control section. All glassware is cleaned per SOP NC-QA-014. Specific selection of reagents may be required to avoid introduction of contaminants.

4.2. Interferences are eliminated or reduced by using the distillation procedure. Oxyidizing agents, such as chlorine, will decompose most cyanides. Sulfide will distill over with the cyanide and could affect colorimetric, titrimetric, and electrode procedures. Refer to the preparation section on how to screen and treat samples appropriately. The possibility of interferences from nitrate and nitrite is eliminated by pretreatment with sulfamic acid just before distillation.

4.3. Aldehydes convert cyanide to cyanohydrin which could result in the loss of cyanide. If the presence of aldehydes are suspected, stabilize the sample with NaOH at the time of collection and add 2 mL 3.5% ethylenediamine solution per 100 mL of sample.

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4.4. Refer to Standard Methods for further information on possible interferences and associated treatments.

5. SAFETY

5.1. Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual, the Facility Addendum to the Corporate EH&S Manual, and this document.

5.2. In the event the sample begins to react unexpectedly during distillation, remove entire apparatus from heat source, set aside, and allow to cool**. DO NOT ATTEMPT TO DISASSEMBLE GLASSWARE.** Doing so may result in a sudden release of pressure with spraying of the sample.

5.3. Ensure cooling water is turned on to the distillation unit. Otherwise, the samples may boil over and come into contact with the heating plates.

5.4. Latex, vinyl, nitrile, or similar gloves may be used.

5.5. Preparation of sodium hydroxide solutions produces considerable amounts of heat. Use plastic containers to mix this solution if possible. If glass containers are used, they must be free of any cracks or irregularities.

5.6. The acidification of samples prior to extraction/preparation can result in the release of a highly toxic gas, hydrogen cyanide.

5.7. If samples are identified with cyanide concentrations equal to or greater than 200 mg/L, immediately notify the Department Manager and personnel responsible for hazardous waste shipping. Those samples must be identified as extremely hazardous for other chemists and must receive special attention during disposal. **Potassium cyanide and sodium cyanide will give off Hyrdogen Cyanide (HCN) gas if combined with strong acids. Inhalation of CN gas can cause irritation, dizziness, nausea, unconsciousness and potentially death.**

5.8. Cyanide and cyanide salts are extremely toxic. Addition of acid can generate hydrogen cyanide gas, which can be extremely dangerous.

5.9. The following is a list of the materials used in this method, which have a serious or significant hazard rating. **NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the Reagents and Standards section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

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Material (1)	Hazards	Exposure Limit (2)	Signs and Symptoms of Exposure
Potassium Cyanide	Poison Corrosive	5 mg/m^3 TWA as CN	**This material will form Hydrogen Cyanide (HCN) gas when combined with strong acids. Breathing HCN gas may result in death.** Corrosive to the respiratory tract. May cause headache, weakness, dizziness, labored breathing nausea and vomiting, which can be followed by weak and irregular heart beat, unconsciousness, convulsions, coma and death. Solutions are corrosive to the skin and eyes, and may cause deep ulcers, which heal slowly. May be absorbed through the skin, with symptoms similar to those noted for inhalation. Symptoms may include redness, pain, blurred vision, and eye damage.
Pyridine	Flammable Irritant	5 ppm-TWA	Inhalation causes severe irritation to the respiratory tract. Symptoms of overexposure include headache, dizziness, nausea, and shortness of breath. Causes severe irritation possibly burns, to the skin. Symptoms include redness and severe pain. Absorption through the skin may occur, resulting in toxic effects similar to inhalation. May act as a photosensitizer. Vapors cause eye irritation. Splashes cause severe irritation, possible corneal burns and eye damage.
Acetic Acid (1)	Corrosive Poison Flammable	10 ppm-TWA	Contact with concentrated solution may cause serious damage to the skin and eyes. Inhalation of concentrated vapors may cause serious damage to the lining of the nose, throat, and lungs. Breathing difficulties may occur.
Acetone	Flammable	1000 ppm-TWA	Inhalation of vapors irritates the respiratory tract. May cause coughing, dizziness, dullness, and headache.
Cadmium Carbonate	Probable carcinogen	0.01 mg/ m^3 as Cd	Ingestion causes increased salivation, choking, vomiting, stomach pains and diahrrea. Inhalation may cause respiratory irritation, nausea and dyspnea.
Sodium Hydroxide	Corrosive Poison	2 ppm, 5 mg/m^3 2 mg/m^3-Ceiling	This material will cause burns if comes into contact with the skin or eyes. Severe irritant. Effects from inhalation of dust or mist vary from mild irritation to serious damage of the upper respiratory tract, depending on severity of exposure. Symptoms may include sneezing, sore throat or runny nose. Contact with skin can cause irritation or severe burns and scarring with greater exposures. Causes irritation of eyes, and with greater exposures it can cause burns that may result in permanent impairment of vision, even blindness.
Potassium Phosphate	Flammable	None	Inhalation causes severe irritation to the respiratory tract. Causes severe irritation possibly burns, to the skin. Symptoms include redness and severe pain. .

Sulfuric Acid	Corrosive Oxidizer Dehydrator Poison Carcinogen	1 mg/m^3-TWA	Inhalation produces damaging effects on the mucous membranes and upper respiratory tract. Symptoms may include irritation of the nose and throat, and labored breathing. Symptoms of redness, pain, and severe burn can occur. Contact can cause blurred vision, redness, pain and severe tissue burns. Can cause blindness.
Barbituric Acid	Irritant	Not established	Limited information. Inhalation may irritate respiratory tract. Causes skin and eye irritation. Must be treated as potential health hazard; do not ingest.
Potassium Hydroxide	Poison Corrosive Reactive	2 mg/m^3 Ceiling	Inhalation symptoms may include coughing, sneezing, damage to the nasal or respiratory tract. High concentrations can cause lung damage. Swallowing may cause severe burns of mouth, throat and stomach. Other symptoms may include vomiting, diarrhea. Severe scarring of tissue and death may result. Contact with skin can cause irritation or severe burns and scarring. Causes irritation of eyes with tearing, redness, and swelling. Greater exposures cause severe burns with possible blindness.
Sodium Cyanide	Poison Corrosive	5 mg/m^3 TWA as CN (skin)	**This material will form Hydrogen Cyanide (HCN) gas when combined with strong acids. Breathing HCN gas may result in death.** Corrosive to the respiratory tract. May cause headache, weakness, dizziness, labored breathing nausea and vomiting, which can be followed by weak and irregular heartbeat, unconsciousness, convulsions, coma and death. Solutions are corrosive to the skin and eyes, and may cause deep ulcers, which heal slowly. May be absorbed through the skin, with symptoms similar to those noted for inhalation. Symptoms may include redness, pain, blurred vision, and eye damage.
Chloramine T Hydrate	Poison		May be harmful by inhalation, ingestion, or skin absorption. This material is irritating to mucous membranes and upper respiratory tract. Avoid contact and inhalation.
Zinc Acetate	Irritant	None Listed	Symptoms of skin or eye contact include redness, itching and pain.
Silver Nitrate	Corrosive Poison Oxidizer	0.01 mg/m^3 (TWA) for silver metal dust and fume as 0.02 Ag	This is a corrosive, poisonous material. It will cause burns to any area of contact and is harmful if inhaled. Ingestion may cause death. Contact with other material may cause fire. Inhalation symptoms may include burning sensation, coughing, wheezing, laryngitis, shortness of breath, headache, nausea, and vomiting. May be absorbed into the body following inhalation. Swallowing can cause severe burns of the mouth, throat and stomach. Can cause sore throat, vomiting, and diarrhea. Poison. Symptoms include pain and burning in the mouth, blackening of the skin and mucous membranes, throat, and abdomen, salivation, vomiting of black material, diarrhea, collapse, shock, coma and death. Skin contact can cause redness, pain and severe burns. Eye contact can cause blurred vision, redness, pain, severe tissue burns and eye damage.
1 – Always add acid to water to prevent violent reactions.			

2 – Exposure limit refers to the OSHA regulatory exposure limit.

5.10 Eye protection that protects against splash, laboratory coat, and appropriate gloves must be worn while samples, standards, solvents, and reagents are being handled. Cut-resistant gloves must be worn doing any other task that presents a strong possibility of getting cut. Disposable gloves that have been contaminated must be removed and discarded; other gloves must be cleaned immediately.

5.11 Exposure to chemicals must be maintained **as low as reasonably achievable.** All samples with stickers that read "Caution/Use Hood!" **must** be opened in the hood. Contact the EH&S Coordinator if this is not possible. Solvent and waste containers must be kept closed unless transfers are being made.

5.12 The preparation of standards and reagents must be conducted in a fume hood with the sash closed as far as the operation will permit.

5.13 All work must be stopped in the event of a known or potential compromise to the health and safety of a TestAmerica associate. The situation must be reported **immediately** to the EH&S Coordinator and Laboratory Supervisor.

6. EQUIPMENT AND SUPPLIES

6.1. Cyanide Distillation Apparatus

6.2. Analytical balance, capable of accurately weighing ± 0.0001 g

6.3. Vacuum pump

6.4. Graduated cylinders: 50 mL

6.5. Volumetric flasks: 100 mL, 200 mL

6.6. Volumetric pipettes: range from 0.01 to 20 mL

6.7. Balance: Top loading, capable of accurately weighing ± 0.01 g

6.8. Lead Acetate Indicator Paper

6.9. Potassium Iodide (KI) Indicator Paper

6.11. Buret: Class A 10 mL

6.12. pH strips

6.13. Beakers: various

6.14. Snap seal containers: 120 mL

6.15. Plastic bottles with lids: 250mL or 500mL

7. REAGENTS AND STANDARDS

7.1. Reagents

7.1.1. Sulfamic Acid: reagent grade

7.1.2. Sulfamic Acid Solution: Add 100 g of sulfamic acid to 800 mL reagent water and dilute to 1 liter with reagent water

7.1.3. Ascorbic Acid: reagent grade

7.1.4. Sodium Hydroxide: (NaOH), high purity grade.

7.1.6. Sodium Hydroxide, 0.25 N: Add 10 g of NaOH to 900 mL reagent water and dilute to 1 liter with reagent water. Purchased reagent may also be used.

7.1.7. Sulfuric Acid: (H_2SO_4), concentrated

7.1.8. Acetic Acid Solution: Add 200 mL of reagent water to a 1.0 L volumetric flask. Carefully add 750 mL of glacial acetic acid, and bring to volume with reagent water. Transfer to a 1.0 L amber bottle and cap.

7.1.9. Magnesium Chloride: ($MgCl_2 \bullet 6H_2O$), reagent grade

7.1.10. Magnesium Chloride Solution: Add 510 g $MgCl_2 \bullet 6H_2O$ to 500 mL reagent water and dilute to 1 liter with reagent water. Purchased reagent may also be used.

7.1.11. Calcium Hypochlorite: [Ca $(OC1)_2$], reagent grade

7.1.12. Calcium Hypochlorite Solution: Add 5 g of $Ca(OC1)_2$ to 100 mL of reagent water. Store in an amber glass bottle in the dark. Prepare monthly.

7.1.13. Methyl Red Indicator: Add 0.05 g of methyl red to 50 mL of glacial acetic acid and dilute to 100 mL with reagent water.

7.1.14. Methyl red reagent grade glacial

7.1.15. Acetic Acid: (CH_3COOH), glacial reagent grade

7.1.16. Zinc Acetate: [$Zn(C_2H_3O_2)$], reagent grade

7.1.17. Zinc Acetate Solution: Add 100 g zinc acetate to 800 mL reagent water and dilute to 1 liter with reagent water.

7.1.18. Sodium Acetate: [$NaC_2H_3O_2 \bullet 3H_2O$] reagent grade

7.1.19. Sodium Acetate Buffer: Add 410 g of sodium acetate to 500 mL of reagent water. Adjust the pH to 4.5 using glacial acetic acid and dilute to 1 liter with reagent water.

7.1.20. Rhodanine: reagent grade

7.1.21. 0.0192 N Silver Nitrate: reagent grade

7.1.23. Cadmium carbonate [$CdCO_3$]: reagent grade

7.2. Standards

7.2.1. Primary Source Cyanide Stock Standard, 1000 mg/L: Add 2.51 g of potassium cyanide (KCN) and 2.0 g of potassium hydroxide (KOH) to a 1000 mL volumetric flask and dilute to volume with reagent water. Mix well and store in glass amber container. Stable for 1-3 months. Additional information can be found in SOP NC-QA-017.

Note: This stock standard may also be purchased.

Note: This stock standard must be standardized prior to use. See SOP NC-WC-031 Appendix I

7.2.2. Secondary Source Cyanide Standard, 1000 mg/L: Follow Section 7.2.1 using an alternate source of Potassium Cyanide (KCN).

Note: This stock standard may also be purchased.

Note: This stock standard must be standardized prior to use. See SOP NC-WC-031 Appendix I.

7.2.3. Calibration Standards (Water and Solid Matrices)

7.2.3.1 If using the purchased 0.25N NaOH: Pipette the appropriate amount of cyanide standard into 100 mL volumetric, and bring to volume with 0.25N NaOH. Prepare weekly.

Concentraction CN-	ML CN-	Final Volume
10 mg/L	10 mL of 100 mg/L	100 mL
1.0 mg/L	20 mL of 10 mg/L	200 mL

8. SAMPLE PRESERVATION AND STORAGE

8.1. Solid and liquid samples are not chemically preserved. Water samples are preserved with NaOH to a pH>12. All samples are stored at 4° C ± 2°C in plastic or glass containers.

8.2. The holding time for samples is 14 days from sampling to analysis.

9. QUALITY CONTROL

9.1. Batch Definition

9.1.1. A batch is a group of no greater than 20 samples excluding QC samples (LCS, Method Blank, MS, MSD) which are processed similarly with respect to the procedure. All sample setups must be initiated within a 24-hour period from the initial preparation or extraction and without interruption of the process. All samples within the batch must be treated with the same lots of reagents and the same process.

9.2. Method Blank (MB)

9.2.1. One method blank (MB) must be processed with each preparation batch. The method blank consists of reagent water containing all reagents specific to the method that is carried through the entire analytical procedure, including preparation and analysis. The method blank is used to identify any system and process interferences or contamination of the analytical system that may lead to the reporting of elevated analyte concentrations or false positive data. The method blank must not contain any analyte of interest at or above the reporting limit.

9.2.1.1. A method blank consists of 50 mL 0.25N NaOH for Total Cyanide analysis or 50 mL reagent water for Weak Acid Dissociable and Amenable Cyanide analysis. The method blank for solids consists of 50 mL 0.25N NaOH or reagent H_2O and 1.0g Ottawa sand. The blank must be distilled per Section 11.4.3 and analyzed with each analytical batch of samples.

9.2.2 Corrective Action for Blanks

9.2.2.1. If the analyte level in the method blank exceeds the reporting limit for the analytes of interest in the sample, all associated samples are reprepared and re-analyzed. If this is not possible due to limited sample quantity or other considerations, the corresponding sample data **must be addressed in the**

project narrative.

9.2.2.2. If there is no analyte greater than the RL in the samples associated with an unacceptable method blank, the data may be reported with qualifiers. **Such action must be addressed in the project narrative.**

9.3. Laboratory Control Sample (LCS)

9.3.1. One LCS must be processed with each preparation batch. The LCS must be carried through the entire analytical procedure. The LCS is used to monitor the accuracy of the analytical process. Ongoing monitoring of the LCS results provides evidence that the laboratory is performing the method within acceptable accuracy and precision guidelines.

9.3.2. A mid-range LCS consisting of a 0.04 mg/L (2.0 mL of 1.0 mg/L secondary source to 50 mL) must be distilled per Section 11.4.3 and analyzed with each analytical batch of samples for Total, Amenable, and Weak Acid Dissociable Cyanide analysis.

Note: A purchased complex cyanide solution may be used instead as the midrange LCS for **Total Cyanide** analysis only.

9.3.3. Corrective Action for Laboratory Control Samples

9.3.3.1. If any analyte is outside established control limits, the system is out of control and corrective action must occur.

9.3.3.2. Corrective action must be repreparation and re-analysis of the batch unless the client agrees that other corrective action is acceptable. For Ohio VAP samples, the LCS must be redistilled, unless the LCS is biased high and the samples are non-detect.

9.3.3.2 The only exception is that if the LCS recoveries are biased high and the associated sample is ND for the parameter(s) of interest, the batch is acceptable. **This must be addressed in the project narrative.**

9.4 Additional information on QC samples can be found in QA Policy QA-003.

9.5 Matrix Spike/Matrix Spike Duplicate (MS/MSD)

9.5.1 One MS/MSD pair must be processed for each batch. A matrix spike (MS) is a field sample to which known concentrations of target analytes have been added. A matrix spike duplicate (MSD) is a second aliquot of the same sample (spiked identically as the MS) prepared and analyzed along with the sample and matrix spike. Some client-specific data quality objectives (DQOs) may require the use of sample duplicates in place of or in addition to MS/MSDs. The MS/MSD results are used to determine the effect of a matrix on the precision and accuracy of the analytical process. Due to the potential variability of the matrix of each sample, these results may have immediate bearing only on the specific

sample spiked. Samples identified as field blanks cannot be used for MS/MSD analysis.

9.5.1.1 An MS/MSD consisting of 50 mL or 1.0g of sample brought up to 50 mL with reagent water and 2.0 mL of the 1.0 mg/L of secondary standard must be distilled with each analytical batch of samples.

9.5.2 Corrective action for MS/MSDs

9.5.2.1 If the analyte recovery or RPD falls outside the acceptance range, the recovery of that analyte must be in control for the LCS. If the LCS recovery is within limits, then the laboratory operation is in control and the results may be accepted. If the recovery of the LCS is outside limits, corrective action must be taken. Corrective action must include repreparation and re-analysis of the batch.

9.5.2.2 If the native analyte concentration in the MS/MSD exceeds four times the spike level for that analyte, the recovery data is reported as MSB and NC for the exception code. The A-spike is not needed in this case.

9.5.2.3 If an MS/MSD is not possible due to limited sample volume, then a laboratory control sample duplicate (LCSD) must be analyzed. The RPD of the LCS and LCSD must be compared to the in-house limits.

9.6 Control Limits

9.6.1 Control limits are established by the laboratory as described in SOP NC-QA-018.

9.6.2 Laboratory control limits are internally generated and updated periodically unless method specified. Control limits are easily accessible via LIMs (QC Browser program).

9.7 Method Detection Limits (MDLs) and MDL Checks

9.7.1 MDLs and MDL Checks are established by the laboratory as described in SOPs NC-QA-021 and CA-Q-S-006.

9.7.2 MDLs are easily accessible via LIMs (QC Browser program).

9.8 Nonconformance and Corrective Action

9.8.1 Any deviations from QC procedures must be documented as a nonconformance, with applicable cause and corrective action approved by the facility QA Manager.

10. CALIBRATION AND STANDARDIZATION

10.1 A low and high standard are distilled from the same source as the calibration curve each day. Prepare the low standard (0.025 mg/L) by diluting 0.125 mL of 10 ppm standard with 0.25N

NaOH to a final volume of 50 mL. Prepare the high standard (0.1 mg/L) by diluting 0.5 mL of 10 ppm standard with 0.25N NaOH to a final volume of 50 mL. If distilled standards do not agree within +10% of the undistilled standards, the analyst must find the cause of the apparent error before proceeding. The instrument is calibrated daily using the 0.2 ppm standard, which is diluted by the instrument into the following concentrations: 0.005, 0.010, 0.025, 0.050, 0.100, 0.200.

11. PROCEDURE

11.1 One-time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, chemistry, sample size, or other parameters. Any variation in procedure must be completely documented using a Nonconformance Memo and is approved by the QA Manager. The Nonconformance Memo must be filed in the project file. This is not applicable for Ohio EPA/VAP.

11.4. Sample Preparation Procedure applicable for use with the Midi distillation unit.

11.4.1 For DoD work, refer to SOP NC-QA-016 for specific details.

11.4.2 Checking for Interferences

11.4.2.1. Using pH paper strips, check the pH of the sample and record it as >12 or <12 on the analytical logsheet. If pH is <12, the deviation **must be addressed in the project narrative.**

11.4.2.2. Test each sample for the presence of sulfides using lead acetate paper. If sulfides are present, treat the sample with powdered cadmium carbonate. Yellow cadmium sulfide precipitates if the sample contains sulfide. Repeat this operation until a drop of the treated sample solution does not darken the lead acetate test paper. Filter the solution. Avoid a large excess of cadmium carbonate and long contact time in order to minimize loss by complexation or occlusion of cyanide on the precipitated material. Document this on the analytical logsheet.

11.4.2.3 Test each sample for the presence of chlorine using potassium iodide test strips. If chlorine is present, add ascorbic acid, a few crystals at a time, until a drop of sample produces no color on the indicator paper. Document this on the analytical logsheet.

11.4.3 Amenable Cyanide (Chlorinated Aliquot)

11.4.3.1. Place 50 mL (waters) or 1.0 g (solids/liquids) into a beaker or snap cap container. Add 50 mL of reagent water to non-waters. Place the beaker on a stir plate and begin mixing. Test the pH of the solution. If less than 12, add 1.25 N NaOH, drop by drop until pH $\geq$ 12. Drop by drop, add calcium hypochlorite until an excess of chlorine is reached. Test for chlorine excess

using KI paper. Allow the sample to chlorinate for one hour maintaining excess chlorine and pH between 11 and 12. If necessary, add more Ca(OCL)2 and/or NaOH. Either cover beakers with aluminum foil, or place box over breakers to keep them in the dark during the chlorination process. At the end of the chlorination period, add about 0.1 to 0.5 g of ascorbic acid to destroy excess chlorine. Test using KI paper. Keep the addition of ascorbic acid to a minimum. Pour the sample into a distillation flask and follow the total cyanide preparation method (Section 11.4.3). Also set up an unchlorinated aliquot of sample (50 mL or 1.0 g) following the total cyanide method.

11.4.4 Total Cyanide

11.4.4.1 Add 50 mL or 1.0 g of the sample to the distillation tube. Bring the final volume to 50 mL with reagent water. Add 50 mL of 0.25 N NaOH solution to the absorber tube and assemble the scrubber. Assemble the cyanide distillation apparatus.

11.4.4.2 Turn on cold flow. Turn on the vacuum source and adjust the flow such that even stream of air bubbles are in the scrubber tube approximately $^{1}/_{4}$ " of foam or 1-2 bubbles per second. At this time, add any spiking solutions to the LCS or MS/MSDs samples directly into the inlet tube.

11.4.4.3 In the order stated, add 2.0 mL sulfamic acid solution, 2.5 mL of concentrated sulfuric acid, and 2.0 mL of magnesium chloride solution to the distillation tube. Be sure to rinse the inlet tube sparingly with reagent water between and after reagent additions.

11.4.4.4 Flip the heater switch to "on" to allow the apparatus to warm up. Be sure to adjust the air flow and water as necessary. Heat for one hour. Allow to cool for 15 minutes. Keep the vacuum and cooling water on.

11.4.4.5 Disconnect the absorber. Pour solution into a 120 mL snap seal container. Check the pH of the solution to ensure it is >12. **NOTE:** If the pH is not >12, do NOT remove the sample from the hood. Contact the Technical Director, Operations Manager, or EH&S representative immediately for further instruction. Do not rinse the scrubber tube or dilute NaOH in the snap seal. Be sure to properly label the bottles "total" or "amenable" along with sample ID, position, and date.

11.4.5 Weak Acid Dissociable

11.4.5.1 Add 50 mL or 1.0 g of sample to the distillation tube. Bring all volumes up to 50 mL with reagent water. Add 50 mL of 0.25 N NaOH to the absorber tube and assemble.

11.4.5.2 Turn on cooling water. Turn on the vacuum source. Add any spiking standards

to the appropriate LCS or MS/MSDs at this time. Through the inlet tube, add 1.0 mL of sodium acetate buffer, 1.0 mL of zinc acetate, and 0.25 mL of methyl red indicator. Rinse the inlet tube sparingly with reagent water between and after reagent additions. If the sample is not red, carefully add 75% acetic acid dropwise until the sample does turn red. Check the sample color periodically throughout the distillation hour to ensure the sample stays red.

Reminder: Do not use the complex purchased cyanide solution standard as the LCS for Weak Acid Dissociable cyanide analysis.

11.4.5.3 Flip on the heater switch (this allows the apparatus to warm up). Adjust the air flow and water as needed. Heat for one hour. Allow the sample to cool for 15 minutes with the air and water on.

11.4.5.4 Pour the scrubber contents into a 120 mL snap seal bottle. Do not rinse the scrubber. Label the bottle well, and be sure to denote it is a Weak Acid Dissociable sample distillate.

11.4.6 Cyanide Unit Cleanup

11.4.6.1 Cyanide distillation unit glassware is very fragile and expensive. It must be handled with care at all times.

11.4.6.2 Disassemble each setup. Be sure to collect the solids in a screen and dispose of properly.

11.4.6.3 Wash each setup with hot water. Rinse several times with reagent water.

Note: After preparing samples found to be extremely high in cyanide, distill 0.25 N NaOH through the system.

.11.4.6.4 Re-assemble the setup.

11.4.6.5 Be sure to wash each setup as a separate unit, and replace in the same position.

11.4.6.6 If a sample of known high cyanide concentration was distilled in a certain position, be sure to change the appropriate tubing on that position.

11.5 Analytical Documentation

11.5.1 Record all analytical information in the analytical logbook/logsheet, including the analytical data from standards, blanks, LCSs, MS/MSDs, and any corrective actions or modifications to the method.

11.5.2 All standards are logged into a department standard logbook. All standards are assigned a unique number for identification. Logbooks are reviewed by the supervisor or designee.

11.5.3 Documentation such as all associated instrument printouts (final runs, screens, reruns, QC samples, etc.) and daily calibration data corresponding to all final runs is available for each data file.

11.5.4 Sample results and associated QC are entered into LIMs after final technical review.

12. DATA ANALYSIS AND CALCULATIONS

12.1. Not applicable

13. METHOD PERFORMANCE

13.1. Each laboratory must have initial demonstration of performance data on file and corresponding method detection limit files.

13.2. Training Qualifications

13.2.1. The Group/Team Leader has the responsibility to ensure this procedure is performed by an associate who has been properly trained in its use and has the required experience.

13.2.2. Method validation information (where applicable) in the form of laboratory demonstrations of capabilities is maintained for this method in the laboratory QA files.

14. POLLUTION PREVENTION

14.1. It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage, and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention".

15. WASTE MANAGEMENT

15.1 All waste must be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees must abide by this method and the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention".

15.2 Waste Streams Produced by the Method

15.2.1 The following waste streams are produced when this method is carried out.

15.2.1.1 Acidic waste. This waste is disposed of in the designated container labeled "Acid Waste".

15.2.1.2 Standard Waste and High Concentration Samples: This waste is disposed of in the designated container labeled "High Cyanide/Basic Waste." NO ACID is added to this container.

15.3. Laboratory personnel assigned to perform hazardous waste disposal procedures must have a working knowledge of the established procedures and practices of TestAmerica. They must have training on the hazardous waste disposal practices upon initial assignment to these tasks, followed by an annual refresher training.

16. REFERENCES

16.1. References.

16.1.1. SW846, Test Methods for Evaluating Solid Waste, Third Edition, Total and Amenable Cyanide, Automated UV; Method 9012A

16.1.2. EPA 600; Cyanide, Total and Cyanide, Amenable to Chlorination; Methods 335.1, 335.4, 335.2, and 335.2(CLP-M)

16.1.3. Standard Methods for the Examination of Water and Wastewater, Eighteenth Edition; Weak Acid Dissociable Cyanide; Method 4500-CN-I

16.1.4. Standard Methods for the Examination of Water and Wastewater, Eighteenth Edition; Complex Cyanide; Method 4500-CN-E, 4500 CN-G

16.1.5. TestAmerica North Canton Quality Assurance Manual (QAM), current version

16.1.7 TestAmerica Corporate Environmental Health and Safety Manual, CW-E-M-001, and TestAmerica North Canton Facility Addendum and Contingency Plan, current version

16.1.8 Corporate Quality Management Plan (CQMP), current version

16.1.9 Revision History

Historical File:		Revision 7.0: 03/19/98		Revision 8.4: 12/08/04
		Revision 8.0: 02/04/99		Revision 8.5: 05/30/08
		Revision 8.1: 03/20/00		Revision 8.6: 12/30/08
		Revision 8.2: 05/31/01		
		Revision 8.3: 06/17/03		

16.2. Associated SOPs and Policies, current version

16.2.1. QA Policy, QA-003

16.2.2 Glassware Washing, NC-QA-014

16.2.3 Statistical Evaluation of Data and Development of Control Charts, NC-QA-018

16.2.4 Method Detection Limits and Instrument Detection Limits, NC-QA-021 and CA-Q-S-006

16.2.5 Supplemental Practices for DoD Projects, NC-QA-016

16.2.5 Standards and Reagents, NC-QA-017

16.2.6 Cyanide, Automated Method, NC-WC-031

17. MISCELLANEOUS (TABLES, APPENDICES, ETC.)

17.1. Method Deviation [9012A, 335.1, 335.2, 335.2(CLP-M) Ohio VAP only]

17.1.1. The reflux distillation apparatus used is the midi distillation.

17.1.2. The volume of sample used is reduced to 50 mL vs. 500 mL using the midi-distillation apparatus.

17.1.3. Method of Standard Addition is not performed for samples with matrix interference (sulfides).

17.1.4. Solid and waste samples are analyzed according to 335.2(CLP-M).

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Title: CYANIDE AUTOMATED, PYRIDINE-BARBITURIC ACID METHOD

[Method: SW846 Method 9012A, EPA Methods 335.2, 335.2 (CLP-M), 335.4, and Standard Methods 4500-CN-E, 4500-CN-I, and 4500-CN-G]

Approvals (Signature/Date):

Technology Specialist	05/26/10 Date	Health & Safety Coordinator	05/25/10 Date
Quality Assurance Manager	05/256/10 Date	Laboratory Director	05/25/10 Date

This SOP was previously identified as SOP No. NC-WC-031, Rev 8.2, dated 12/30/08

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1. SCOPE AND APPLICATION

1.1. This method is applicable to the determination of Total, Amenable, and Weak Acid Dissociable Inorganic Cyanide in solids, liquids, and waters. It is based on SW846 Method 9012A, 335.2, 335.2(CLP-M), 335.4 and Standard Methods 4500-CN-E, 4500-CN-I, and 4500-CN-G. The working linear range is 0.005 – 0.2 mg/L for waters and 0.25-10 mg/kg for solids. Reporting limits are listed in Section 17.

1.2. This document accurately reflects current laboratory Standard Operating Procedures (SOP) as of the date above. All facility SOPs are maintained and updated as necessary.

1.3. The associated method codes are SW846 Method 9012A (QP), EPA 335.1(CF), 335.2 (CG), 335.2(CLP-M) (14), EPA 335.4 (O2), Standard Method 4500-CN-E (A4), and Standard Methods 4500 CN-I (HF) and 4500-CN-G (8O).

2. SUMMARY OF METHOD

2.1. The Cyanide, as HCN, is released by distilling/refluxing the sample with strong acid and is trapped in a sodium hydroxide solution.

2.2. The sodium hydroxide solution is analyzed colormetrically on an autoanalyzer using the pyridine-barbituric acid method.

3. DEFINITIONS

3.1. Refer to the glossary in the TestAmerica North Canton Quality Assurance Manual (QAM), current version.

4. INTERFERENCES

4.1. Method interferences may be caused by contaminants in solvents, reagents, glassware, and other processing apparatus that lead to discrete artifacts. All of these materials must be routinely demonstrated to be free from interferences under conditions of the analysis by running laboratory method blanks as described in the Quality Control section. All glassware is cleaned per SOP NC-QA-014. Specific selection of reagents may be required to avoid introduction of contaminants.

4.2. Sulfides interfere, but can be eliminated by treating the sample with cadmium carbonate prior to analysis.

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5. SAFETY

5.1. Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual, the Facility Addendum to the Corporate EH&S Manual, and this document.

5.2. The following is a list of the materials used in this method, which have a serious or significant hazard rating. **NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the Reagents and Standards section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Potassium Cyanide	Poison Corrosive	5 mg/m^3 TWA as CN	**This material will form Hydrogen Cyanide (HCN) gas when combined with strong acids. Breathing HCN gas may result in death.** Corrosive to the respiratory tract. May cause headache, weakness, dizziness, labored breathing nausea and vomiting, which can be followed by weak and irregular heart beat, unconsciousness, convulsions, coma and death. Solutions are corrosive to the skin and eyes, and may cause deep ulcers, which heal slowly. May be absorbed through the skin, with symptoms similar to those noted for inhalation. Symptoms may include redness, pain, blurred vision, and eye damage.
Pyridine	Flammable Irritant	5 ppm-TWA	Inhalation causes severe irritation to the respiratory tract. Symptoms of overexposure include headache, dizziness, nausea, and shortness of breath. Causes severe irritation possibly burns, to the skin. Symptoms include redness and severe pain. Absorption through the skin may occur, resulting in toxic effects similar to inhalation. May act as a photosensitizer. Vapors cause eye irritation. Splashes cause severe irritation, possible corneal burns and eye damage.
Sodium Hydroxide	Corrosive Poison	2 ppm, 5 mg/m^3 2 mg/m^3-Ceiling	This material will cause burns if comes into contact with the skin or eyes. Severe irritant. Effects from inhalation of dust or mist vary from mild irritation to serious damage of the upper respiratory tract, depending on severity of exposure. Symptoms may include sneezing, sore throat or runny nose. Contact with skin can cause irritation or severe burns and scarring with greater exposures. Causes irritation of eyes, and with greater exposures it can cause burns that may result in permanent impairment of vision, even blindness.

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Potassium Phosphate	Flammable	None	Inhalation causes severe irritation to the respiratory tract. Causes severe irritation possibly burns, to the skin. Symptoms include redness and severe pain. .
Sodium Phosphate		None	Inhalation may cause respiratory tract irritation. Can produce delayed pulmonary edema. Causes mild skin and eye irritation. Injestion may cause gastrointestinal irritation.
Hydrochloric Acid	Corrosive Poison	5 ppm-Ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Cadmimum Carbonate	Probable carcinogen	0.01 mg/ m^3 as Cd	Ingestion causes increated salivation, choking, vomiting, stomach pains and diahrrea. Inhalation may cause respiratory irritation, nausea and dyspnea.
Sulfuric Acid	Corrosive Oxidizer Dehydrator Poison Carcinogen	1 mg/m^3-TWA	Inhalation produces damaging effects on the mucous membranes and upper respiratory tract. Symptoms may include irritation of the nose and throat, and labored breathing. Symptoms of redness, pain, and severe burn can occur. Contact can cause blurred vision, redness, pain and severe tissue burns. Can cause blindness.
Barbituric Acid	Irritant	Not established	Limited information. Inhalation may irritate respiratory tract. Causes skin and eye irritation. Must be treated as potential health hazard; do not ingest.
Potassium Hydroxide	Poison Corrosive Reactive	2 mg/m^3 – Ceiling	Inhalation symptoms may include coughing, sneezing, damage to the nasal or respiratory tract. High concentrations can cause lung damage. Swallowing may cause severe burns of mouth, throat and stomach. Other symptoms may include vomiting, diarrhea. Severe scarring of tissue and death may result. Contact with skin can cause irritation or severe burns and scarring. Causes irritation of eyes with tearing, redness, swelling. Greater exposures cause severe burns with possible blindness.
Chloramine T Hydrate	Poison		May be harmful by inhalation, ingestion, or skin absorption. This material is irritating to mucous membranes and upper respiratory tract. Avoid contact and inhalation.
Zinc Acetate	Irritant	None Listed	Symptoms of skin or eye contact include redness, itching and pain.

Silver Nitrate	Corrosive Poison Oxidizer	0.01 mg/m^3 (TWA) for silver metal dust and fume as 0.02 Ag	This is a corrosive, poisonous material. It will cause burns to any area of contact and is harmful if inhaled. Ingestion may cause death. Contact with other material may cause fire. Inhalation symptoms may include burning sensation, coughing, wheezing, laryngitis, shortness of breath, headache, nausea, and vomiting. May be absorbed into the body following inhalation. Swallowing can cause severe burns of the mouth, throat and stomach. Can cause sore throat, vomiting, and diarrhea. Poison. Symptoms include pain and burning in the mouth, blackening of the skin and mucous membranes, throat, and abdomen, salivation, vomiting of black material, diarrhea, collapse, shock, coma and death. Skin contact can cause redness, pain and severe burns. Eye contact can cause blurred vision, redness, pain, severe tissue burns and eye damage.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

5.3. Ensure cooling water is turned on to the distillation unit. Otherwise, the samples may boil over and come into contact with the heating plates.

5.4. Preparation of sodium hydroxide solutions produces considerable amounts of heat. Use plastic containers to mix this solution, if possible. If glass containers are used, they must be free of any cracks or irregularities.

5.5. The acidification of samples prior to extraction/preparation can result in the release of a highly toxic gas--hydrogen cyanide.

5.6. If samples are identified with cyanide concentrations equal to or greater than 200 mg/L, immediately notify the Department Manager and personnel responsible for hazardous waste shipping. Those samples must be identified as extremely hazardous for other chemists and must receive special attention during disposal.

5.7. Potassium cyanide and sodium cyanide will give off hyrdogen cyanide (HCN) gas if combined with strong acids. Inhalation of CN gas can cause irritation, dizziness, nausea, unconsciousness, and potentially death.

5.8. Cyanide and cyanide salts are extremely toxic. Addition of acid can generate hydrogen cyanide gas, which can be extremely dangerous.

5.9. Eye protection that protects against splash, laboratory coat, and appropriate gloves must be worn while samples, standards, solvents, and reagents are being handled. Cut-resistant gloves must be worn doing any other task that presents a strong possibility of getting cut. Disposable

gloves that have been contaminated must be removed and discarded. Other gloves must be cleaned.

5.10. Exposure to chemicals must be maintained **as low as reasonably achievable.** All samples with stickers that read "Caution/Use Hood!" **must** be opened in the hood. Contact the EH&S Coordinator if this is not possible. Solvent and waste containers must be kept closed unless transfers are being made.

5.11. It is recommended that neat standards be purchased only as a last resort. The preparation of standards from neat materials and reagents must be conducted in a fume hood with the sash closed as far as the operations will permit.

5.12. Standards in solution may be diluted in the open laboratory when syringes and the like are utilized.

5.13. All work must be stopped in the event of a known or potential compromise to the health and safety of a TestAmerica associate. The situation must be reported **immediately** to the EH&S Coordinator and to a laboratory supervisor.

6. EQUIPMENT AND SUPPLIES

6.1. 2 mL and 4mL Cuvettes

6.2. 100 mL, 250 mL, 1000 mL volumetric flasks

6.3 Volumetric pipettes: various, ranging from 0.01 mL to 20 mL

6.4 Top loading balance: capable of accurately weighing ± 0.01g

6.5 Balance: Analytical, capable of accurately weighing 0.0001g

7. REAGENTS AND STANDARDS

7.1. Reagents

7.1.1. Cadmium carbonate: powder

7.1.2. Phosphate buffer: Add 136 g of potassium phosphate - monobasic (KH_2PO_4) and 2.8 g of sodium phosphate – dibasic anhydrous (Na_2HPO_4) to 800 mL of reagent water in a 1 liter volumetric flask. Mix, bring to volume with reagent water (may be purchased commercially.)

7.1.3. Chloramine-T reagent: Add 1.0 g of chloramine-T to a 250 mL volumetric flask and dilute to volume with reagent water. Prepare fresh daily. (May be purchased commercially.)

7.1.4. Pyridine reagent: Add 15.0 g of barbituric acid to a 1 liter volumetric flask. Add 75 mL of pyridine and 15 mL of concentrated hydrochloric acid (HCl) and mix. Bring to volume with reagent water and store at 4°C ± 2°C in an amber glass bottle. The maximum shelf life is six months or the vendor's expiration date, whichever is earlier

Note: The pyridine barbituric acid may be purchased commercially. Filter 50 ml of the pyridine barbituric acid, and bring up to 250 ml with DI water.

7.1.5. 0.25 N sodium hydroxide: Add 250 mL of 1N NaOH to a 1 liter volumetric flask, and dilute to volume with reagent water.

Note: 0.25 N NaOH may be purchased instead.

7.1.6. Rhodanine indicator, purchased.

7.1.7. 0.0192 N silver nitrate, purchased

7.2. Standards

7.2.1. Primary Source Cyanide Stock Standard, 1000 mg/L: Add 2.51 g of potassium cyanide (KCN) and 2.0 g of potassium hydroxide (KOH) to a 1000 mL volumetric flask and dilute to volume with reagent water. Mix well and store in glass amber container. Stable for 1-3 months. Additional information can be found in SOP NC-QA-017.

Note: This stock standard may also be purchased.

Note: This stock standard must be standardized prior to use. See Appendix I.

7.2.2. Secondary Source Cyanide Standard, 1000 mg/L: Follow Section 7.2.1 using an alternate source of Potassium Cyanide (KCN).

Note: This stock standard may also be purchased.

Note: This stock standard must be standardized prior to use. See Appendix I.

7.2.3. Calibration Standards (Water and Solid Matrices)

7.2.3.1. Pipette the appropriate amount of cyanide standard into 100 mL volumetric

and bring to volume with 0.25N NaOH. The low standard must be at, or below, the reporting limit. Prepare weekly.

Concentration CN- Calibration Standards	ML CN-	Final Volume
10 mg/L	1 mL of 1000 mg/L	100 mL Dilution from stock primary and secondary
1.0 mg/L (secondary only)	20 mL of 10 mg/L	200 mL LCS/MS
0.1 mg/L (secondary only)	1 mL of 10 mg/L	100 mL ICV
0.005 mg/L	0.05 mL of 10 mg/L	100 mL MRL check
* 0.2 mg/L	2 mL of 10 mg/L	100 mL Calibrant
0.1 mg/L	1 mL of 10 mg/L	100 mL CCV Solution

*Denotes calibration standards

8. SAMPLE COLLECTION, PRESERVATION, AND STORAGE

8.1. Solid samples are not chemically preserved. Water samples are preserved with NaOH to a pH >12. All samples are stored at 4°C ± 2°C in plastic or glass containers.

8.2. The holding time for samples is 14 days from sampling to analysis.

9. QUALITY CONTROL

9.1. Batch Definition

9.1.1. A batch is a group of no greater than 20 samples, excluding QC samples (LCS, Method Blank, MS, MSD), which are processed similarly with respect to the procedure. All sample setups must be initiated within a 24-hour period from the initial preparation or extraction and without interruption of the process. All samples within the batch must be treated with the same lots of reagents and the same processes.

9.2. Method Blank

9.2.1. One method blank (MB) must be processed with each preparation batch. The method blank consists of reagent water or 0.25N NaOH containing all reagents specific to the method that is carried through the entire analytical procedure, including preparation and analysis. The method blank is used to identify any system and process interferences or contamination of the analytical system that may lead to the reporting of elevated analyte

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concentrations or false positive data. The method blank must not contain any analyte of interest at, or above, the reporting limit. For Ohio VAP Projects, the method blank must not contain any analyte above the reporting limit.

9.2.2. A method blank consists of 50 mL 0.25N NaOH for Total Cyanide analysis or 50 mL reagent water for Weak Acid Dissociable and Amenable Cyanide analysis must be distilled and analyzed with each analytical batch of samples. See SOP NC-WC-032 for distillation instructions.

9.2.3. Corrective Action for Blanks

9.2.3.1. If the analyte level in the method blank exceeds the reporting limit for the analytes of interest in the sample, all associated samples are reprepared and re-analyzed. If this is not possible due to limited sample quantity or other considerations, the corresponding sample data **must be addressed in the project narrative.**

9.2.3.2. If there is no analyte greater than the RL in the samples associated with an unacceptable method blank, the data may be reported with qualifiers. **Such action must be addressed in the project narrative.**

9.3. Laboratory Control Sample (LCS)

9.3.1. One LCS from an independent source must be processed with each preparation batch. The LCS must be carried through the entire analytical procedure. The LCS is used to monitor the accuracy of the analytical process. Ongoing monitoring of the LCS results provides evidence that the laboratory is performing the method within acceptable accuracy and precision guidelines.

9.3.2. A midrange LCS consisting of a 0.04 mg/L (2.0 mL of 1.0 mg/L of the secondary source to 50 mL) must be distilled and analyzed with each analytical batch of samples for Total, Amenable, and Weak Acid Dissociable Cyanide analysis. See SOP NC-WC-032 for distillation instruction.

Note: A purchased complex cyanide solution may be used instead as the midrange LCS for Total Cyanide analysis only.

9.3.3. Corrective Action for LCS

9.3.3.1. If any analyte is outside established control limits, the system is out of control and corrective action must occur.

9.3.3.2. Corrective action will be reprepparation and re-analysis of the batch unless the client agrees that other corrective action is acceptable. For Ohio VAP

samples, the LCS must be redistilled, unless the LCS is biased high and the samples are non-detect.

9.3.3.3. The only exception is if the LCS recoveries are biased high and the associated sample is ND for the parameter(s) of interest, the batch is acceptable. **This must be addressed in the project narrative.**

9.4. Additional information on QC samples can be found in QA Policy QA-003.

9.5. Matrix Spike/Matrix Spike Duplicate (MS/MSD)

9.5.1. One MS/MSD pair must be processed for each batch. A matrix spike (MS) is a field sample to which known concentrations of target analytes have been added. A matrix spike duplicate (MSD) is a second aliquot of the same sample (spiked identically as the MS) prepared and analyzed along with the sample and matrix spike. Some client-specific data quality objectives (DQOs) may require the use of sample duplicates in place of, or in addition to, MS/MSDs. The MS/MSD results are used to determine the effect of a matrix on the precision and accuracy of the analytical process. Due to the potential variability of the matrix of each sample, these results may have immediate bearing only on the specific sample spiked. Samples identified as field blanks cannot be used for MS/MSD analysis.

9.5.2. An MS/MSD consisting of 50 mL or 1.0 g sample and 0.04 mg/L spike (2.0 mL of 1.0 mg/L to 50 mL) must be distilled and analyzed with every batch. See SOP NC-WC-032 for distillation instructions.

9.5.3. Corrective action for MS/MSDs

9.5.3.1. If the analyte recovery or RPD falls outside the acceptance range, the recovery of that analyte must be in control for the LCS. If the LCS recovery is within limits, then the laboratory operation is in control and the results may be accepted. If the recovery of the LCS is outside limits, corrective action must be taken. Corrective action must include repreparation and reanalysis of the batch.

9.5.3.2. If the native analyte concentration in the MS/MSD exceeds four times the spike level for that analyte, the recovery data is reported as "amount" MSB. The Exception Code is changed to NC. The A-spike is not needed in this case.

9.5.3.3. If an MS/MSD is not possible due to limited sample volume, then a laboratory control sample duplicate (LCSD) must be analyzed. The RPD of

the LCS and LCSD must be compared to the matrix spike RPD limits. The LCSD recovery is compared to the LCS recovery limits.

9.6. QC Acceptance Criteria

9.6.1. Control limits are established by the laboratory as described in NC-QA-018.

9.6.2. Laboratory control limits are internally generated and updated periodically unless method specified. The latest version is easily accessible via LIMs (QC Browser program.).

9.7. Method Detection Limits (MDLs) and MDL Checks

9.7.1. MDLs and MDL Checks are established by the laboratory as described in SOPs CA-Q-S-006 and NC-QA-021.

9.7.2. MDLs are easily accessible via LIMs (QC Browser program).

9.8. Nonconformance and Corrective Action

9.8.1. Any deviations from QC procedures must be documented as a nonconformance with applicable cause and corrective action approved by the facility QA Manager.

10. CALIBRATION AND STANDARDIZATION

10.1. Initial Calibration

10.1.1 The instrument is calibrated daily using the 0.2 ppm standard, which is diluted by the instrument into the following concentrations: 0.005, 0.010, 0.025, 0.050, 0.100, 0.200. The calibration is verified by using a midrange ICV. The ICV is composed of the 0.1 ppm secondary standard. The ICV must not vary from the original curve by more than ± 10%, or recalibration is required. The correlation coefficient of the original curve must be $\geq$ 0.995, or recalibration is required. The curve must not be forced through the origin. An ICB sample is analyzed after the ICV. It cannot contain the analyte of interest above the reporting limit or recalibration is required.

10.1.1.1. Linear Regression

The linear fit uses the following functions:

$$y = ax + b$$

or

$$x = \frac{(y - b)}{a}$$

Where: y = Instrument response
x = Concentration
a = Slope
b = Intercept

10.2. Continuing Calibration

10.2.1. The run is checked every ten samples and at the end of the run using a midrange CCV to verify continued linearity. It cannot vary from the original curve by more than ± 10%, or recalibration is required. The previous ten samples must be re-analyzed and bracketed by a CCV that passes criteria. The CCV is composed of the 0.1 mg/L primary standard.

10.2.2. System cleanliness is checked every ten samples and at the end of the run using a CCB. It cannot contain the analyte of interest above the reporting limit or recalibration is required. The previous ten samples must be re-analyzed and bracketed by a CCB that passes criteria. The CCB is 0.25N NaOH.

10.3. High and Low Standard

10.3.1. The distillation technique is checked by distilling a high and low standard and comparing the values obtained to the standard curve. The method recommends that the HI/LO standards be compared to the curve with a +/-10% agreement. Re-analysis must occur if the standards do not meet criteria. The HI/LO standards are evaluated against all applicable batch QC.

11. PROCEDURE

11.1. One-time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, chemistry, sample size, or other parameters. Any variation in procedure must be completely documented using a Nonconformance Memo and is approved by a Technical Specialist and QA Manager. The Nonconformance Memo must be filed in the project file.

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11.2. Any unauthorized deviations from this procedure must also be documented as a nonconformance with a cause and corrective action described. This is not applicable for Ohio EPA/VAP.

11.3. Sample Preparation

11.3.1. See Cyanide Distillation SOP NC-WC-032.

11.3.1.1. The sample is distilled/refluxed under acidic conditions for one hour. The released HCN is trapped in 50 mL of 0.25 N NaOH solution. For EPA Method 335.4, reflux for one and a half hours.

11.3.2. Sample Preparation Procedure

11.3.2.1 All Solid Samples: Test each sample for the presence of sulfides using lead acetate paper. If sulfides are present, treat the sample with powdered cadmium carbonate. Yellow cadmium sulfide precipitates if the sample contains sulfide. Repeat this operation until a drop of the treated sample solution does not darken the lead acetate test paper. Filter the solution. Avoid a large excess of cadmium carbonate and long contact time in order to minimize loss by complexation or occlusion of cyanide on the precipitated material.

Note: Water must be tested prior to distillation. See Cyanide Distillation SOP NC-WC-032.

11.3.3 For DoD work, refer to SOP NC-QA-016 for specific details.

11.4. Sample Analysis

11.4.1. Recommended Instrument Conditions

11.4.1.1. See manufacturer's information for operation instructions.

11.4.1.2. Perform instrument startup.

11.4.1.3. Add reagents to the Konelab reagent wheel.

11.4.1.4. Insert standards and samples to autosampler segments.

11.4.2. Sample Analysis Procedure

11.4.2.1. See manufacturer's information for operating instructions.

11.4.2.2. The correlation coefficient must be > 0.995 to continue. If new standards were not made, then the previous curve may be used if all QC passes criteria.

11.4.2.3. The ICV (from the secondary source) and the ICB is analyzed at the beginning of every run followed by an MRL check. CCVs (from the primary source) and CCBs are analyzed at the end and between every ten samples.

11.4.2.4. Sample distillates higher than the highest calibration standard (0.2 mg/L) must be diluted with 0.25 N NaOH and re-analyzed.

11.4.2.5. Any samples analyzed after a high sample must be re-analyzed if carryover is suspected.

11.4.2.6. If the sample concentration exceeds the working range as defined by the calibration standards, then the sample must be diluted and re-analyzed. Dilutions must target the most concentrated analyte in the upper half (over 50% of the high level standard) of the calibration range. It may be necessary to dilute samples due to matrix.

11.5. Analytical Documentation

11.5.1. Record all analytical information in the analytical logbook/logsheet, including the analytical data from standards, blanks, LCSs, MS/MSDs, and any corrective actions or modifications to the method.

11.5.2. All standards and reagents are logged into a department standard logbook. All standards are assigned a unique number for identification. Logbooks are reviewed by the supervisor or designee.

11.5.3. Documentation such as all associated instrument printouts (final runs, screens, reruns, QC samples, etc.) and daily calibration data corresponding to all final runs is available for each data file.

11.5.4. Sample results and associated QC are entered into LIMs after final technical review.

12. DATA ANALYSIS AND CALCULATIONS

12.1. $$\textit{Total Cyanide, mg / L} = \frac{\textit{mg / L } CN^{-} \quad \textit{x 50}}{\textit{mL of sample distilled}} \textit{ x D}$$

12.2. $$\textit{Total Cyanide, mg / kg} = \frac{\textit{mg / L } CN^{-} \quad \textit{x 50}}{\textit{g of sample distilled.}} \textit{ x D}$$

12.3. $$\textit{Amenable Cyanide, mg / L} = \textit{Total } CN^{-} \textit{ (mg / L) - Chlorinated } CN^{-} \textit{ (mg / L)}$$

Where:

mg/L = can also be mg/kg

D = Dilution Factor = $\frac{\textit{Final Volume of Dilution}}{\textit{Volume of Sample Distillate Used}}$

Note: Weak Acid Dissociable Cyanide has the same calculations as Total Cyanide

12.4. LCS Recovery:

$$\frac{\textit{Instrument Value}}{\textit{0.04 (true)}} \textit{ x 100} = \textit{\% Recovery}$$

Note: The true value may vary by manufacturer or analysis.

12.5. CCV Recovery:

$$\frac{\textit{Instrument Value}}{\textit{0.1 (true)}} \textit{ x 100} = \textit{\% Recovery}$$

NOTE: *CCV recovery must be between 90-110% for data to be acceptable. If CCV recovery is not within these limits, re-analysis is required.*

12.6. MS/MSD Recovery for Waters and solids

$$\frac{\textit{A - B}}{\textit{0.040 (true)}} \textit{ x 100} = \textit{\% Recovery}$$

Where:

A = Instrument value MS/MSD

B = Sample instrument value

12.7 Additional equations and calculations are listed in the following SOPs: Calibration Curves (General), CA-Q-S-005, and Selection of Calibration Points, CA-T-P-002.

13. METHOD PERFORMANCE

13.1. Each laboratory must have initial demonstration of performance data on file and corresponding method detection limit files.

13.2. Training Qualifications

13.2.1. The Group/Team Leader has the responsibility to ensure this procedure is performed by an associate who has been properly trained in its use and has the required experience.

13.2.2. Method validation information (where applicable) in the form of laboratory demonstrations of capabilities is maintained for this method in the laboratory QA files.

14. POLLUTION PREVENTION

14.1. It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage, and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention".Waste Management

15. WASTE MANAGEMENT

15.1. All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention".

15.2. Waste Streams Produced by the Method

15.2.1. The waste from Konelab analysis is collected in the waste bucket housed in the instrument. The waste container is emptied at the end of the day in the "Acid Waste" container.

15.2.2. Filter paper contaminated with cadmium sulfide complex. This waste is placed in a container labeled "Solid Waste".

15.2.3. Aqueous rinsates from distillation tube clean up. This waste is collected in the lab and disposed of in a container labeled "Acid Waste".

15.2.4. Standard Waste and High Concentration Samples: This waste is disposed of in the designated container labeled "High Cyanide/Basic Waste." NO ACID is added to this container.

15.3. Laboratory personnel assigned to perform hazardous waste disposal procedures must have a working knowledge of the established procedures and practices of TestAmerica. They must have training on the hazardous waste disposal practices upon initial assignment to these tasks followed by annual refresher training.

16. REFERENCES

16.1. References

16.1.1. SW846, Test Methods for Evaluating Solid Waste Method 9012A

16.1.2. EPA 600; Cyanide, Total and Cyanide, Amenable to Chlorination; Methods 335.2, March 1983

16.1.3. EPA 600; Determination of Total Cyanide by Semi-Automated Colorimetry, 335.4, Revision 1.0, August 1993

16.1.4. Standard Methods for the Examination of Water and Wastewater, Eighteenth Edition: Weak and Dissociable Cyanide; Method 4500-CN-E, 4500-CN-I, and 4500-CN-G

16.1.5. TestAmerica North Canton Quality Assurance Manual (QAM), current version

16.1.6. TestAmerica Corporate Environmental Health and Safety Manual, CW-E-M-001, and TestAmerica North Canton Facility Addendum and Contingency Plan, current version

16.1.7. Corporate Quality Management Plan (CQMP), current version

16.1.8. Revision History

Historical File:		Revision 5: 06/24/99		Revision 8.1: 05/30/08
		Revision 6: 06/28/99		Revision 8.2: 12/30/08
		Revision 7: 05/31/01		
		Revision 8: 11/08/04		

16.2. Associated SOPs and Policies, latest version

16.2.1. QA Policy, QA-003

16.2.2. Glassware Washing, NC-QA-014

16.2.3. Statistical Evaluation of Data and Development of Control Charts, NC-QA-018

16.2.4. Method Detection Limits and Instrument Detection Limits, NC-QA-021 and CA-Q-S-006

16.2.5. Supplemental Practices for DoD Project Work, NC-QA-016

16.2.6. Cyanide Preparation Method, NC-WC-032

16.2.7. Standards and Reagents, NC-QA-017

16.2.8. Selection of Calibration Points, CA-T-P-002

16.2.9. Calibration Curves (General), CA-Q-S-005

17. MISCELLANEOUS (TABLES, APPENDICES, ETC.)

17.1. Reporting limits

17.1.1. The reporting limit (RL) is 0.01 mg/L for waters (50 mL used) and 0.50 mg/kg for solids (1.0 g used). The lowest level of the calibration curve can be used as the reporting limit upon request.

17.1.2. If samples require dilution or smaller volumes than specified in this method, the RL will be elevated.

17.2. Method Deviation (9012A/335.2)

17.2.1. Method of Standard Addition is not performed for samples with matrix interference (sulfides).

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Appendix I

CYANIDE STANDARDIZATION

1. Pipette 10.0 mL of the 1000 ppm stock cyanide standard into a 250 mL Erlenmeyer flask and add 90 mL of reagent water.

2. Add 0.5 mL (10 drops) of Rhodanine indicator.

3. Titrate with 0.0192 N silver nitrate (using a micro burette) until the color changes from yellow to pink/orange.

4. Titrate a blank (100 mL reagent water) following Steps 2 and 3.

5. Calculation

$$\textbf{Cyanide, mg / L} = \frac{\textbf{(A - B) (1000)}}{\textbf{mL Cyanide Solution (10)}}$$

Where:
A = mL titrant for standard
B = mL titrant for blank

6. If the cyanide concentration is not 1000 ppm, adjust concentration accordingly.

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Title: EXTRACTION OF ORGANIC COMPOUNDS FROM WATERS AND SOILS, BASED ON SW846 3500 SERIES, AND 600 SERIES METHODS

Approvals (Signature/Date):

Technology Specialist	Date	Health & Safety Coordinator	Date
Quality Assurance Manager	Date	Laboratory Director	Date
Technical Director	Date		

This SOP was previously identified as SOP No. NC-OP-032, Rev 0, dated 03/12/08

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1. SCOPE AND APPLICATION

1.1 This SOP describes procedures for preparation (extraction) of semivolatile organic analytes in aqueous, TCLP leachate, and soil matrices for analysis by Gas Chromatography (GC) and Gas Chromatography / Mass Spectrometry (GC/MS). The procedures are based on SW846 and 600 series methodology and are applicable for measurements made to comply with the Resource Conservation and Recovery Act (RCRA) and for wastewater testing.

1.1.1. Extraction procedures for the following determinative methods are covered: 8081A, 8082, 8270C, 8015B, 608, and 625

1.1.2. The extraction procedures here may be appropriate for other determinative methods when appropriate spiking mixtures are used.

1.1.3. Other extraction procedures (PFE, SPE, etc.) may be used, but are not currently covered in this SOP.

1.1.4. The applicable LIMS method codes are QJ (8081A), QL (8270C), QH (8082), DM (608), DP (625), KI (8015B).

2. SUMMARY OF METHOD

2.1. Separatory Funnel Extraction

A measured volume of sample, typically 1 liter, is adjusted, if necessary, to a specified pH and serially extracted with methylene chloride using a separatory funnel.

2.2. Continuous Liquid/Liquid Extraction

A measured volume of sample, typically 1 liter, is placed into a continuous liquid/liquid extractor, adjusted if necessary, to a specific pH and extracted with the appropriate solvent for 18-24 hours.

2.3. Sonication Extraction

A measured weight of sample, typically 30 g, is mixed with anhydrous sodium sulfate until free flowing. This is solvent extracted three times using an ultrasonic horn.

2.4. Soxhlet Extraction (Accelerated and Traditional)

A 30 g sample is mixed with anhydrous sodium sulfate until free flowing. This is extracted with refluxing solvent.

2.5. Concentration

Procedures are presented for drying and concentration of the extract to final volume for analysis.

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3. DEFINITIONS

3.1. Definitions of terms used in this SOP may be found in the glossary of the TestAmerica North Canton Quality Assurance Manual (QAM), current version.

4. INTERFERENCES

4.1. Method interferences may be caused by contaminants in solvents, reagents, glassware, and other processing apparatus. All these materials must be routinely demonstrated to be free from interferences under conditions of the analysis by running laboratory method blanks as described in the Quality Control section. Specific selection of reagents may be required to avoid introduction of contaminants.

4.2. Visual interferences or anomalies (such as foaming, emulsions, odor, etc.) must be documented.

5. SAFETY

5.1. Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual and this document.

5.2. Eye protection that protects against splash, laboratory coat and appropriate gloves must be worn while samples, standards, solvents and reagents are being handled. Cut-resistant gloves must be worn doing any other task that presents a strong possibility of getting cut. Disposable gloves that have become contaminated must be removed and discarded, other gloves must be cleaned immediately.

5.3. The following analytes have been tentatively classified as known or suspected, human or mammalian carcinogens: benzo(a)anthracene, benzidine, 3,3'dichlorobenzindine, benzo(a)pyrene, alpha-BHC, beta-BHC, gamma-BHC, delta-BHC, dibenz(a,h)anthracene, N-nitrosodimethylamine, 4,4'-DDT, and polychlorinated biphenyl compounds. Primary standards of these toxic compounds must be prepared in the hood.

5.4. The following is a list of the materials used in this method, which have a serious or significant hazard rating. NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table. A complete list of materials used in the method can be found in the Reagents and Standards section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

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Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Methylene Chloride	Carcinogen Irritant	25 ppm-TWA 125 ppm-STEL	Causes irritation to respiratory tract. Has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting and headache. Causes irritation, redness and pain to the skin and eyes. Prolonged contact can cause burns. Liquid degreases the skin. May be absorbed through skin.
Sodium Hydroxide	Corrosive Poison	2 ppm, 5 mg/m^3 2 Mg/M3-Ceiling	This material will cause burns if comes into contact with the skin or eyes. Severe irritant. Effects from inhalation of dust or mist vary from mild irritation to serious damage of the upper respiratory tract, depending on severity of exposure. Symptoms may include sneezing, sore throat or runny nose. Contact with skin can cause irritation or severe burns and scarring with greater exposures. Causes irritation of eyes, and with greater exposures it can cause burns that may result in permanent impairment of vision, even blindness.
Sulfuric Acid (1)	Corrosive Oxidizer Dehydrator Poison Carcinogen	1 mg/m^3	This material will cause burns if comes into contact with the skin or eyes. Inhalation of vapors will cause irritation of the nasal and respiratory system. Symptoms may include irritation of the nose and throat, and labored breathing. Symptoms of redness, pain, and severe burn can occur. Contact can cause blurred vision, redness, pain, and severe tissue burns. Can cause blindness.
Acetone	Flammable	1000 ppm-TWA	Inhalation of vapors irritates the respiratory tract. May cause coughing, dizziness, dullness, and headache.
Hydrochloric Acid	Corrosive Poison	5 ppm-Ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Acetonitrile	Flammable Poison	40 ppm-TWA	Early symptoms may include nose and throat irritation, flushing of the face, and chest tightness. Prolonged exposure to high levels of vapors may cause formation of cyanide anions in the body.
Hexane	Flammable Irritant	500 ppm-TWA	Inhalation of vapors irritates the respiratory tract. Overexposure may cause lightheadedness, nausea, headache, and blurred vision. Vapors may cause irritation to the skin and eyes.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

5.5. Exposure to hazardous chemicals must be maintained **as low as reasonably achievable.** All samples with stickers that read "Caution/Use Hood!" **must** be opened in the hood. Contact

the EH&S Coordinator if this is not possible. Solvent and waste containers must be kept closed unless transfers are being made.

5.6. The preparation of standards and reagents and glassware cleaning procedures that involve solvents such as methylene chloride must be conducted in a fume hood with the sash closed as far as the operations will permit. If more than 500 mL of methylene chloride is spilled, evacuate the area until the area has been cleaned by EH&S.

5.7. All work must be stopped in the event of a known or potential compromise to the health and safety of a TestAmerica North Canton associate. The situation must be reported **immediately** to the EH&S Coordinator and the Laboratory Supervisor.

5.8. During Kuderna-Danish (KD) concentration, do not allow the extract to boil to dryness. The solvent vapors remaining in the KD apparatus may superheat and create an explosion or fire hazard. The KD apparatus and glass separatory funnels have ground glass joints which can become stuck. Technicians must use Kevlar or other cut/puncture-resistant gloves when separating stuck joints.

5.9. 3510 Separatory Funnel

5.9.1. The use of separatory funnels to extract aqueous samples with methylene chloride creates excessive pressure very rapidly. Initial venting must be done immediately after the sample container has been sealed and inverted. Periodic venting may be necessary during the extraction. Vent the funnel into the hood away from people and other samples. This is considered a high-risk activity. The use of a face shield over safety glasses or goggles is recommended. Keep the sash on the fume hood as low as reasonably possible.

5.10. 3520 Extraction Continuous Liquid/Liquid

5.10.1. All personnel are to ensure liquid-liquid area is clear of unnecessary items. Heating mantles used with liquid-liquid extractions generate temperatures that could ignite some materials that come in contact with the heating mantles.

5.10.2. Ensure all solvents are away from liquid-liquid extractor. Increased temperatures near solvents can cause the pressure in the containers to increase.

5.10.3. Ensure all boiling flasks have cooled to room temperature before disconnecting liquid-liquid bodies from boiling flasks to prevent any burns.

6. EQUIPMENT AND SUPPLIES

6.1. Glassware must be cleaned per Glassware Washing, SOP NC-QA-014.

6.2. Equipment and supplies for extraction procedures:

EQUIPMENT AND SUPPLIES	Sep Fun.	CLLE	Soni	Sox	Conc
Separatory Funnel: 2 L	Ö				
Separatory Funnel Rack	Ö				
Balance: >1400 g capacity, accurate ±1 g	Ö	Ö	Ö	Ö	
pH indicator paper, Ranges: 0-14, 7.5-14, 0-6, and 3.8-5.5	Ö	Ö			
Graduated Cylinder: 1 liter. (other sizes may be used as needed)	Ö	Ö			Ö
Erlenmeyer Flask: 125 & 300 mL (other sizes optional)	Ö	Ö	Ö		Ö
Centrifuge	Ö				
Methylene Chloride Collection Tank	Ö	Ö			
Initial Volume Template	Ö	Ö			
Solvent Dispenser Pump or 100 mL Graduated Cylinder	Ö	Ö	Ö	Ö	Ö
Continuous Liquid/Liquid Extractor		Ö			
Round or flat Bottom: 250, 500 mL or 1 L		Ö		Ö	
Boiling Chips: Contaminant free, approximately 10/40 mesh (Teflon® PTFE, carbide or equivalent)		Ö		Ö	Ö
Cooling Condensers		Ö		Ö	
Heating Mantle: Rheostat controlled		Ö		Ö	
Auto-timer for heating mantle		Ö		Ö	
Beakers: 250 & 400 mL, graduated	Ö	Ö	Ö		Ö
450mL wide-mouth glass jars	Ö		Ö		Ö
Balance: >100 g capacity, accurate ±0.1 g	Ö	Ö	Ö	Ö	Ö
Soxhlet Extractor				Ö	
Cellulose and Glass Thimbles				Ö	
Accelerated Soxhlet Extractor (Soxtherm-trade name)				Ö	
Sonicator (at least 300 watts)			Ö		
Sonicator horn, 3/4 inch			Ö		
Micro-tip sonicator horn			Ö		
Kuderna-Danish (K-D) Apparatus: 500 mL					Ö
Concentrator Tube: 10 mL, attached to K-D with clips					Ö
Snyder Column: Three-ball macro					Ö
Water Bath: Heated, with concentric ring cover, capable of temperature control (± 5°C) up to 95°C. The bath must be used in a hood or with a solvent recovery system.					Ö
Vials: Glass, 2 mL, 4 mL, 10 mL, and 40 mL capacity with Teflon®-lined screw-cap					Ö
Nitrogen Blowdown Apparatus					Ö
Nitrogen: reagent grade.					Ö
Culture Tubes: 10 mL, 16 mmx100 mm					Ö
Syringe: 1 mL	Ö	Ö	Ö	Ö	
Glass Wool	Ö	Ö	Ö	Ö	
Glass Funnel: 75 X 75 mm	Ö	Ö	Ö	Ö	Ö
Disposable Pipettes, 5 ¾ in, and 9in.	Ö	Ö	Ö	Ö	Ö
Aluminum Foil	Ö	Ö	Ö	Ö	Ö
Paper Towels	Ö	Ö	Ö	Ö	Ö

6. REAGENTS AND STANDARDS

7.1. Reagents for Extraction Procedures

All reagents must be ACS reagent grade or better unless otherwise specified.

REAGENTS	Sep Fun.	CLLE	Soni	Sox	Conc
Sodium hydroxide (NaOH), Pellets: Reagent Grade	Ö	Ö			
Sodium hydroxide solution, 10 N: Dissolve 40 g of NaOH in reagent water and dilute to 100 mL.	Ö	Ö			
Sulfuric acid (H_2SO_4), Concentrated: Reagent Grade	Ö	Ö			
Sulfuric acid (1:1): Carefully add 500 mL of H_2SO_4 to 500 mL of reagent water. Mix well.	Ö	Ö			
Hydrochloric Acid (HCl)			Ö		
Organic free reagent water.	Ö	Ö			
Sodium sulfate (Na_2SO_4), Granular, Anhydrous: Purify by heating at 400°C a minimum of two hours.	Ö	Ö	Ö	Ö	Ö
Magnesium sulfate			Ö	Ö	
Extraction/Exchange Solvents: Methylene chloride, hexane, acetonitrile, acetone, pesticide quality or equivalent	Ö	Ö	Ö	Ö	Ö
Acetone, Methylene Chloride: Used for cleaning	Ö	Ö	Ö	Ö	Ö

7.2. Standards

7.2.1. Stock Standards

Stock standards are purchased as certified solutions. Semivolatile stock standards are stored at $\leq 6^{o}$C. All stock standards must be protected from light. Stock standard solutions must be replaced after one year (from the time of preparation, if prepared in house, or from the time the ampoule is opened if purchased). Standards must be allowed to come to room temperature before use.

7.2.2. Surrogate Spiking Standards

Prepare or purchase surrogate spiking standards at the concentrations listed in Table 5. Surrogate spiking standards are purchased or prepared as dilutions of the stock standards. Surrogate spiking solutions must be refrigerated and protected from light. The standards must be replaced at least every six months or sooner if there is reason to believe that the standard has degraded or concentrated.

7.2.3. Matrix Spiking and Laboratory Control Spiking Standards

The same spiking solution is used for the matrix spike and the Laboratory Control Sample. Prepare MS/LCS spiking standards at the concentrations listed in Table 6. Spiking standards are purchased or prepared as dilutions of the stock standards.

Spiking solutions must be refrigerated and protected from light. The standards must be replaced at least every six months or sooner if there is reason to believe that the

standard has degraded or concentrated.

7.2.4 See SOP NC-QA-017 for additional information on Standards and Reagents.

8. SAMPLE COLLECTION PRESERVATION AND STORAGE

8.1. Samples are not chemically preserved.

8.2. Samples are stored at 4°C ± 2°C in glass containers with Teflon®-lined caps.

8.3. Holding Times

8.3.1 The holding time for aqueous samples is seven days from sampling to extraction. The holding time for solid and waste samples is 14 days from sampling to extraction.

8.3.2 For TCLP leachates, the holding time is 14 days from sampling to the leach process. The extraction holding time seven days from when the TCLP Leach tumbling has been completed, excluding the filtration step, to the extraction step.. If the filtration step requires extended times, this time counts as part of the seven-day holding time.

8.3.3. Analysis of the extracts is completed within 40 days of extraction.

9. QUALITY CONTROL

9.1. Quality Control Batch

9.1.1. The batch is a set of up to 20 samples that are of the same matrix and are processed together using the same procedures and reagents. The batch must contain a method blank, an LCS, and a matrix spike / matrix spike duplicate. (In some cases, at client request, it may be appropriate to process a matrix spike and sample duplicate in place of the MS / MSD). If clients specify specific samples for MS/MSD, the batch may contain multiple MS/MSD. See Policy QA-003 for further definition of the batch.

9.2. Insufficient Sample

9.2.1. If insufficient sample is available to process a MS/MSD, then a second LCS must be processed. The LCS pair is then evaluated according to the LCS/LCSD criteria. Use of an LCS pair in place of an MS/MSD must be documented.

9.3. Sample Count

9.3.1. Laboratory-generated QC samples (method blanks, LCS, MS/MSD) are not included in the sample count. Field samples are included.

9.4. Method Blank

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9.4.1. A method blank consisting of all reagents added to the samples must be prepared and analyzed with each batch of samples. Surrogates are spiked into the method blank at the same level as the samples. See Table 3 for the appropriate amount of surrogate to use for each analytical method. The method blank is used to identify any background interference or contamination of the analytical system which may lead to the reporting of elevated concentration levels or false positive data.

9.4.2. Aqueous Method Blanks use 1000 mL of reagent water spiked with the surrogates. The method blank goes through the entire analytical procedure.

9.4.3. Solid method blanks use approximately 30 g of sodium sulfate spiked with the surrogates. See Table 3 for the appropriate amount of surrogate to use for each analytical method. The method blank goes through the entire analytical procedure.

9.4.4. TCLP method blanks use 250 mL of leachate fluid spiked with the surrogates. SPLP method blanks use 1000 mL of leachate fluid spiked with the surrogates The leachate may optionally be diluted to 1000 mL with reagent water. The method blank goes through the entire analytical procedure.

9.5. Laboratory Control Sample (LCS)

9.5.1. Laboratory Control Samples are well-characterized laboratory-generated samples used to monitor the laboratory day-to-day performance of routine analytical methods. The LCS, spiked with a group of target compounds representative of the method analytes, is used to monitor the accuracy of the analytical process, independent of matrix effects. Ongoing monitoring of the LCS results provides evidence that the laboratory is performing the method within accepted QC guidelines for accuracy and precision. The LCS goes through the entire analytical procedure.

9.5.2. The LCS is made up in the same way as the method blank (see sections 9.4.1 through 9.4.4), but spiked with the LCS standard and the surrogates. See Tables 3 and 4 for the appropriate amount of spike to use for each analytical method.

9.6. Surrogates

9.6.1. Surrogates are organic compounds which are similar to the target analyte(s) in chemical composition and behavior in the analytical process, but which are not normally found in environmental samples.

9.6.2. Each applicable sample, blank, LCS, and MS/MSD is spiked with surrogate standards. Surrogate spike recoveries must be evaluated by determining whether the concentration (measured as percent recovery) falls within the required recovery limits. See Table 3 for the appropriate amount of surrogate spike to use for each analytical method.

9.7. Matrix Spike/Matrix Spike Duplicate (MS/MSD)

9.7.1. A matrix spike is an environmental sample to which known concentrations of target analytes have been added. A matrix spike duplicate is a second spiked aliquot of the same sample, which is prepared and analyzed along with the sample and matrix spike. See Tables 3 and 4 for the appropriate amount of spike to use for each analytical method.

9.8. Initial Demonstration of Capability

9.8.1. The initial demonstration and method detection limit studies described in Section 13 must be acceptable before analysis of samples may begin.

9.9 Control Limits

9.9.1 Control limits are established by the laboratory as described in SOP NC-QA-018.

9.9.2 Laboratory control limits are internally generated and updated periodically unless method specified. Control limits are easily accessible via the LIMs (QC Browser program).

9.10 Method Detection Limits (MDLs) and MDL Checks

9.10.1 MDLs and MDL Checks are established by the laboratory as described in SOPs CA-Q-S-006 and NC-QA-021.

9.10.2 MDLs are easily accessible via the LIMs (QC Browser program).

9.11 Nonconformance and Corrective Action

9.11.1 Any deviations from QC procedures must be documented as a nonconformance, with applicable cause and corrective action approved by the facility QA Manager.

10. CALIBRATION AND STANDARDIZATION

10.1. On a weekly basis, measure the appropriate volume of solvent into the appropriate size glass vial using a gastight syringe that is manufactured to a certified volume delivery tolerance of ± 0.01 mL. The "standard" glass vial is sealed, and the meniscus is marked by etching a line on the bottle. The glass vials containing the sample extracts are then compared against the "standard" glass vial to ensure the final volume is consistently 1.0 ± 0.01 mL. A log is kept of the glass vial lot number and preparation date.

11. PROCEDURE

Procedures for separatory funnel liquid/liquid extraction (Section 11.2), continuous liquid/liquid extraction (Section 11.3), sonication extraction (Section 11.4), medium-level sonication (Section 11.5), soxhlet extraction (Section 11.6), accelerated soxhlet (Section 11.7), waste dilution (Section 11.8),

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extract concentration (Section 11.9), and nitrogen evaporation to final concentration (Section 11.10). Refer to SOP NC-QA-016 for information on DoD samples.

11.1. Procedural Variations

Procedural variations are allowed only if deemed necessary in the professional judgment of the supervisor to accommodate variation in sample matrix, chemistry, sample size, or other parameters. Any variation in procedure must be completely documented using a Nonconformance memo and approved by a supervisor and QA/QC Manager. The Nonconformance memo will be filed in the project file. Procedural variations are not allowed for Ohio VAP projects.

11.2. Separatory Funnel Liquid/Liquid Extraction of Water Samples

11.2.1. Remove surrogate and matrix spiking solutions from refrigerator and allow to return to room temperature.

11.2.2. Measure the initial sample pH by inserting a disposable pipette into the sample, and placing a drop of sample on the wide-range pH paper. Record on the extraction benchsheet.

11.2.3. Measure the initial volume using the volume template. Place the template next to the sample bottle and read the volume marking from the template. Record this volume on the benchsheet. The normal sample volume is 1 liter. Other sample volumes may be used to obtain specific reporting limits, and reduced sample volumes, diluted to 1 liter with reagent water, may be used for very dirty samples.

11.2.4. Prepare a method blank, LCS and/or LCSD, and MS/MSD for each batch as specified in Section 9 of this SOP. Use 1 L of reagent water for method blanks and LCS and/or LCSD. Use 500 mL of sample for the MS/MSD. The LCS and/or LCSD and MS/MSD are spiked with the surrogate and matrix spike solutions, the method blank only with the surrogates. See Tables 3 and 4 for the appropriate amount of spike solution to use for each analytical method.

11.2.5. Use 250 mL of leachate for TCLP pesticides and TCLP semivolatiles measured in a beaker. Use 1000 mL of leachate for SPLP semivolatiles and SPLP pesticides.

11.2.6. For a TCLP method blank, LCS and LCS Dup measure 250 mL of the buffer solution used in the leaching procedure and transfer to the separatory funnel. Add 60 mL of methylene chloride to the separatory funnel. The TCLP leachate may be diluted to approximately 1 liter before extraction if needed due to matrix. For an SPLP method blank, LCS, and LCS Dup, measure 1000 mL of the buffer solution using the volume template and transfer to the separatory funnel.

11.2.7. Pour the sample into a separatory funnel. Add 60 mL of methylene chloride per sample. Place a labeled collection jar under each appropriate separatory funnel.

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Place a small amount of glass wool into a funnel and fill with anhydrous sodium sulfate. Place a funnel containing sodium sulfate on each collection jar. Spike the samples with surrogate and/or spike solutions.

11.2.8. Adjust sample pH as indicated in Table 1 for the initial extraction. Use the minimum amount of 1:1 H_2SO_4 or 10 N NaOH, as necessary. Recheck the sample by inserting a disposable pipette into the sample, and placing a drop of sample onto the pH paper. Record adjusted pH, spiking volumes, and standard numbers on the benchsheet. Return spiking solutions to the refrigerator as soon as possible.

11.2.9. Seal and shake or rotate the separatory funnel vigorously for two minutes with periodic venting to release excess pressure. An autoshaker may be used to shake and rotate the separatory funnel.

Warning: Dichloromethane creates excessive pressure very rapidly! Therefore, initial venting must be done immediately after the separatory funnel has been sealed and inverted. Vent into hood away from analysts and other samples.

11.2.10. Allow the organic layer to separate from the water phase until complete visible separation has been achieved. If the emulsion interface between layers is more than one-third the size of the solvent layer, the analyst must employ mechanical techniques to complete the phase separation. The optimum technique depends upon the sample and may include stirring, filtration of the emulsion through glass wool, centrifugation, or other physical methods. If the emulsion cannot be broken (recovery of <80% of the methylene chloride*), transfer the sample, solvent, and emulsion into the extraction chamber of a continuous extractor and proceed as described in continuous liquid-liquid extraction (Section 11.3.). If this is done, the sample must be extracted as part of a valid CLLE batch.

***Note**: 15 - 20 mL of methylene chloride is expected to dissolve in 1 L of water. Thus, solvent recovery could be as low as 35 mL from the first shake and still be acceptable. Subsequent shakes must recover at least 50 mL of solvent.

11.2.11. Drain the solvent extract from the separatory funnel through the prepared filtration funnel into a clean glass container. The extract may be drained directly into the KD flask. Close the stopcock just before the water level begins draining out of the separatory funnel. If the sodium sulfate becomes saturated with water, replace the existing sodium sulfate with fresh drying agent.

11.2.12. Repeat the extraction process two more times using fresh 60 mL portions of solvent, combining the three solvent extracts in the collection container.

11.2.13. If extraction at a secondary pH is required, replace the filtration funnel and adjust the pH of the sample in the separatory funnel to the pH indicated in Table 1 with a minimum amount of 10 N NaOH or 1:1 H_2SO_4. Measure by inserting a disposable pipette into the sample, and placing a drop of sample onto the pH paper. Record the adjusted pH on the benchsheet. Serially extract with three 60 mL portions of methylene chloride, as outlined in Steps 11.2.7 to 11.2.10. Collect these three

extracts in the same container used for the previous fraction.

11.2.14. Rinse the extract residue from the sodium sulfate by pouring 20-30 mL of clean methylene chloride through the funnel and into the collection container.

11.2.15. Dispose of solvent and water remaining in the extractor into the appropriate waste container.

11.2.16. Cover with aluminum foil if the extract is not concentrated immediately. Refer to Section 11.9 for concentration.

11.3. Continuous Liquid/Liquid Extraction from Water Samples

11.3.1. Remove surrogate and matrix spiking solutions from refrigerator and allow to return to room temperature.

11.3.2. Assemble the apparatus. Add approximately 250 mL of methylene chloride to the extractor body. Add three to five boiling chips to the round-bottom distilling flask. Label the flask with an extraction ID label.

11.3.3. Measure the initial sample pH with wide-range pH by inserting a disposable pipette into the sample, and placing a drop of sample onto the wide range pH paper. Record on the extraction benchsheet.

11.3.4. Measure the initial volume using the volume template. Place the template next to the sample bottle and read the volume marking from the template. Record this volume on the benchsheet. Prepare a method blank, LCS, and MS/MSD for each batch as specified in Section 9 of this SOP. See Tables 3 and 4 for the appropriate amount of spike to use for each analytical method. Use 1 L of reagent water for method blanks and LCS.

Use 250mL of leachate for TCLP semivolatiles and TCLP pesticides. Use 1000 mL of leachate for SPLP semivolatiles and SPLP pesticides. Dilute to about 1 liter with reagent water.

For a TCLP method blank, LCS, and LCS Dup, measure 250 mL of the buffer solution in a beaker and transfer to the continuous liquid/liquid extractor. Dilute to about 1 liter with reagent water. For an SPLP method blank, LCS, and LCS Dup, measure 1000 mL of the buffer solution using the volume template and transfer to the continuous liquid/liquid extractor. No dilution with reagent water is required.

Less than one liter of sample may be used for highly contaminated samples, or if the reporting limit can be achieved with less than one liter of sample. In this event, dilute the sample to about 1 liter with reagent water. This must be documented with a Non-Conformance Memo.

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11.3.5. Add reagent water to the extractor body until approximately 150 mL of methylene chloride is pushed over into the round-bottomed flask to ensure proper operation and solvent cycling. Prime the extractor using reagent water. The method blank and samples are spiked with the surrogates, the LCS and matrix spikes with the surrogates, and matrix spiking solutions.

Adjust sample pH as indicated in Table 1 for the initial extraction. Use the minimum amount of 1:1 H_2SO_4 or 10 N NaOH, as necessary. Recheck the sample with pH paper. Record adjusted pH, spiking volumes and standard numbers on the benchsheet. Return spiking solutions to the refrigerator as soon as possible. Attach cold condenser (about 10^{o}C). Turn on heating mantle. Inspect joints for leaks once solvent has begun cycling. Extract for 18-24 hours (24 hours required for 600 series).

If extraction at a secondary pH is required (see Table 1), turn off the heating mantle and allow the extractor to cool. Detach the condenser and adjust the pH of the sample in the extractor body to the pH indicated in Table 1 with a minimum amount of 10 N NaOH or 1:1 H_2SO_4. Measure by inserting a disposable pipette into the sample, and placing a drop of sample on the pH paper. Record the adjusted pH on the benchsheet. Re-attach the condenser, and turn on the heating mantle. Extract for 18-24 hours.

Turn off the heating mantle and allow the extractor to cool.

Cover with aluminum foil if the extract is not concentrated immediately. Refer to Section 11.9 for concentration.

11.4. Sonication

11.4.1. Remove surrogate and matrix spiking solutions from refrigerator and allow to warm to room temperature.

11.4.2. Decant and discard any water layer on a sediment/soil sample, unless there are specific instructions not to decant the water. Record and document if a water layer was discarded on the benchsheet. Homogenize the sample by mixing it thoroughly in the container. If this is not possible, place the sample in clean beaker and homogenize. Upon completion of homogenization in the beaker, return sample to original container. Discard foreign objects such as sticks, leaves and rocks, unless extraction of this material is required by the client. If the sample consists primarily of foreign materials, consult with the client (via the Project Manager or Administrator).

11.4.3. Weigh 30 g of sample ± 0.2g into a 250 or 400 mL beaker or wide-mouth jar. Record the weight to the nearest 0.01 g in the appropriate column on the benchsheet.

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11.4.4. Add appropriate volume of matrix spiking solution to any matrix spike / matrix spike duplicate samples (see Table 4). Add the appropriate volume of the surrogate spiking solution to each sample, method blank, Laboratory Control Sample (LCS), and matrix spikes (see Table 3 for appropriate amounts). Refer to Table 6 for details of the surrogate spiking solutions. Add the appropriate matrix spiking solution to each Matrix Spike/Matrix Spike Duplicate (MS/MSD) and LCS. Refer to Tables 3 and 5 for details of the spiking solutions. Record spiking volumes and standard numbers on the benchsheet. Return spiking solutions promptly to refrigerator.

11.4.5. Mix the weighed and spiked/surrogated sample with a spatula adding enough anhydrous sodium sulfate (approximately 30 g) to be free flowing. (If the sample is not free flowing, extraction efficiency may be reduced)

11.4.6. Prepare a method blank, LCS, and MS/MSD for each batch as specified in Section 9 of this SOP. Use 30 g of sodium sulfate for the method blank and LCS. Use 30 g ± 0.2g each of parent sample for the MS and MSD samples.

11.4.7. Immediately add approximately 100 mL of solvent to the beaker.

Solvents:

Semivolatile GC/MS, TPH, Organochlorine pesticides and PCBs	1:1 v/v Methylene Chloride / Acetone
Semivolatile GC/MS concrete only	Methylene chloride

Note: Steps 11.4.5 through 11.4.9 must be performed rapidly to avoid loss of the more volatile extractables.

11.4.8. Place the bottom surface of the appropriate disrupter horn tip approximately ½ inch below the surface of the solvent, but above the sediment layer.

11.4.9. Sonicate for three minutes, making sure the entire sample is agitated.
Note: Do ***not*** use ***Micro-tip*** probe.

11.4.10. Loosely plug the stem of a 75 mm x 75 mm glass funnel with glass wool. Add 10-20 g of anhydrous sodium sulfate to the funnel cup.

11.4.11. Place the prepared funnel on a labeled collection apparatus (beaker or K-D Apparatus).

11.4.12. Decant and filter extracts through the prepared funnel into the collection apparatus (a clean beaker or K-D Apparatus).

11.4.13. Repeat the extraction two more times with approximately 100 mL portions of solvent each time. Decant off extraction solvent after each sonication. On the final sonication, pour the entire sample (sediment and solvent) into the funnel and rinse

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with an additional 10 mL-20 mL of the appropriate solvent (refer to Table in 11.4.7).

Note: Alternatively, the three extracts may be collected together and then filtered through the sodium sulfate.

11.4.14. Cover with aluminum foil if the extract is not concentrated immediately. Refer to Section 11.9 for concentration

11.4.15. Sonicator Tuning: Tune the sonicator according to manufacturer's instructions. The sonicator must be tuned quarterly and if a new horn is installed.

11.5 Medium level sonication

11.5.1. Remove surrogate and matrix spiking solutions from refrigerator and allow to warm to room temperature.

11.5.2 Decant and discard any water layer on a sediment/soil sample, unless there are specific instructions not to decant the water. Record and document if a water layer was discarded on the benchsheet. Homogenize the sample by mixing it thoroughly in the container. If this is not possible, place the sample in clean beaker and homogenize. Upon completion of homogenization in the beaker, return sample to original container. Discard foreign objects such as sticks, leaves and rocks, unless extraction of this material is required by the client. If the sample consists primarily of foreign materials, consult with the client (via the Project Manager or Administrator).

11.5.3 Weigh 5 g of sample ± 0.1g into a 40 mL vial. Record the weight to the nearest 0.01 g in the appropriate column on the benchsheet.

11.5.4 Add appropriate volume of matrix spiking solution to any matrix spike / matrix spike duplicate samples (see Table 4). Add the appropriate volume of the surrogate spiking solution to each sample, method blank, Laboratory Control Sample (LCS), and matrix spikes (see Table 3 for appropriate amounts). Refer to Table 6 for details of the surrogate spiking solutions. Add the appropriate matrix spiking solution to each Matrix Spike/Matrix Spike Duplicate (MS/MSD) and LCS. Refer to Tables 3 and 5 for details of the spiking solutions. Record spiking volumes and standard numbers on the benchsheet. Return spiking solutions promptly to refrigerator.

11.5.5 Mix the weighed and spiked/surrogated sample with a spatula adding enough anhydrous sodium sulfate to be free flowing.

11.5.6 Prepare a method blank, LCS, and MS/MSD for each batch as specified in Section 9 of this SOP. Use 5 g of sodium sulfate for the method blank and LCS. Use 5 g ± 0.1g each of parent sample for the MS and MSD samples.

11.5.7 Immediately add the appropriate mLs of solvent to the vial.

LCS, MS/MSD	18 mL Hexane
Sample and blank	19 mL Hexane

Note: Steps 11.4.5 - 11.4.9 must be performed rapidly to avoid loss of the more volatile extractables.

11.5.8 Place the bottom surface of the appropriate disrupter horn tip approximately ½ inch below the surface of the solvent, but above the sediment layer.

11.5.9 Sonicate for two minutes continuous disruption, making sure the entire sample is agitated. **Note:** Use ***Micro-tip*** probe.

11.5.10 The samples are now ready for analysis.

11.6 Soxhlet

11.6.1 Remove surrogate and matrix spiking solutions from refrigerator and allow to warm to room temperature.

11.6.2 Decant and discard any water layer on a sediment/soil sample unless there are specific instructions not to decant. Record and document if a water layer was discarded on the benchsheet. Homogenize the sample by mixing it thoroughly in the container. If this is not possible, place the sample in a clean beaker and homogenize. Upon completion of homogenization in the beaker, return the sample to original container. Discard foreign objects such as sticks, leaves, and rocks, unless extraction of this material is required by the client. If the sample consists primarily of foreign materials, consult with the client (via the Project Manager).

11.6.3 Place approximately 200mL of solvent into a 250 mL flat bottom flask containing one or two clean boiling chips. Weigh 30g of sample ± 0.2g into a thimble or in a beaker, recording the weight to the nearest 0.01g on the benchsheet.. Sample weights less than 30g, but over 5g, may be used if the appropriate reporting limits can be met.

11.6.4 Prepare a method blank, LCS, and MS/MSD for each batch as specified in Section 9 of this SOP, using sodium sulfate as the matrix for the LCS. The parent sample is used for the MS/MSD. The weight of sodium sulfate used must be approximately the weight of soil used in each sample.

11.6.5 Add the appropriate amount of surrogate and matrix spiking solution as indicated in Tables 3, 4, 5, and 6.

11.6.6 Add anhydrous sodium sulfate to each sample and mix well. The mixture must have a free-flowing texture. If not, add more sodium sulfate. Add the sample/sodium sulfate mixture to a soxhlet extractor thimble, but do not pack the thimble tightly. The soxhlet extractor or extraction thimble must drain freely for the duration of the extraction

period. A glass wool plug below the sample in the soxhlet extractor is an acceptable alternative for the thimble.

11.6.7 Attach the flask to the extractor and extract the sample for 16-24 hours at 4-6 cycles per hour. Check the system for leaks at the ground glass joints after it has warmed up.

Note: If a reduced quantity of sample is extracted, it is usually necessary to increase the amount of sodium sulfate added or increase the solvent boiling rate to properly set the cycling rate.

Solvents:

Semivolatile GC/MS, OPP, PAH, TPH Organochlorine pesticides and PCBs	1:1 v/v Methylene Chloride / Acetone

11.6.8 Allow the extract to cool after the extraction is complete, then disassemble by gently twisting the soxhlet from the flask.

11.6.9 The sample is now ready for the concentration step (Section 11.9).

11.6.10 Cover with aluminum foil if the extract is not concentrated immediately. Refer to Section 11.9 for concentration.

11.7 Accelerated Soxhlet (Soxtherm Trade Name)

11.7.1 Remove surrogate and matrix spiking solutions from refrigerator and allow to return to room temperature.

11.7.2 Decant and discard any water layer on a sediment/soil sample unless there are specific instructions not to decant. Record and document if a water layer was discarded on the benchsheet. Homogenize the sample by mixing it thoroughly in the container. If this is not possible, place the sample in clean beaker and homogenize. Upon completion of homogenization in beaker, return sample to original container. Discard foreign objects such as sticks, leaves and rocks, unless extraction of this material is required by client. If the sample consists primarily of foreign materials, consult with the client (via the Project Manager or Administrator).

11.7.3 Weigh 30g of sample ± 0.2 g into a beaker or wide-mouth jar, recording the weight to the nearest 0.01g on the benchsheet. Add 30g of anhydrous sodium sulfate to each sample, and mix well. The mixture must have a free-flowing texture. If not, add more sodium sulfate. Add the sample/sodium sulfate mixture to an accelerated soxhlet thimble, but do not pack the thimble tightly. The extraction thimble must drain freely for the duration of the extraction period. A glass wool plug below the sample in the thimble is required.

11.7.4 Sample weights less than 30g, but over 5g, may be used if the appropriate reporting limits can be met.

11.7.5 Prepare a method blank, LCS, and MS/MSD for each batch as specified in Section 9 of this SOP, using sodium sulfate as the matrix. The weight of sodium sulfate used must be approximately the weight of soil used in each sample.

11.7.6 Add the appropriate amount of surrogate and matrix spiking solution as indicated in Tables 3, 4, 5, and 6.

11.7.7 Place thimble in the extract beaker containing at least six clean boiling chips and add approximately 110 mL of solvent. Place beakers into positions on the accelerated soxhlet unit. Run appropriate program for the extraction solvent. Check the system for leaks at the joints periodically.

Solvents:

Semivolatile MS, PAH, TPH Semivolatile GC PCB, PEST, OPP	1:1 v/v Methylene Chloride / Acetone 1:1 v/v Hexane/Acetone

11.7.8 Upon completion of the program, remove the extract beaker from the unit, let cool, and dispose of the extracted sample.

11.7.9 Transfer extract into a culture tube, rinsing the extract beaker to complete the quantitative transfer. Rinse the extractor beaker, which contained the solvent extract with 10-15 mL of the appropriate solvent and pour it through the sodium sulfate drying funnel. Rinse the funnel with 10-15 mL of methylene chloride to complete the quantitative transfer.

11.7.10 Place culture tubes on nitrogen evaporation unit and reduce to approximately 1-2 mL. Add appropriate solvent according to the extraction performed to bring the sample to the correct final volume.

11.8 Waste Dilution

11.8.1 Remove surrogate and matrix spiking solutions from refrigerator and allow to return to room temperature.

11.8.2 Label the vial with the sample number. Tare the vial, then transfer approximately 1g of sample to the vial. Record the weight to the nearest $\pm$ 0.01g.

11.8.3 For the blank and LCS/LCSD, add a small amount of the appropriate solvent to the vial. Add appropriate volume of surrogate and spike solutions (Table 3).

11.8.4 Dilute to 10 mL with the appropriate solvent (Methylene Chloride for GC/MS Semi and GCS TPH). Add 10 mL of appropriate solvent (Hexane) for GCS pesticide and/or PCB analysis. Method 8015B waste dilutions are diluted to approximately 10 mL with DCM and are placed on the nitrogen evaporation unit to reduce to a 2 mL final volume.

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11.8.5 Cap and shake or vortex each extract.

11.8.6 The sample is now ready for analysis.

11.9 Concentration

According to the type of sample, different solvents and final volumes will be required. Refer to Table 2 for the appropriate final volumes and concentrations.

11.9.1 Kuderna-Danish (KD) Method:

11.9.1.1 Assemble a Kuderna-Danish concentrator by attaching a 10 mL concentrator tube to the 500 mL KD flask. Label the CT and KD. Transfer the sample to the labeled K-D flask, filtering Continuous Liquid/Liquid and Soxhlet samples through funnels filled with sodium sulfate. Rinse the funnel with 20-30 mL of methylene chloride to complete the quantitative transfer.

11.9.1.2 Add one or two clean boiling chips and the extract to be concentrated to the KD flask and attach a three-ball Snyder Column. Add approximately 1 mL of clean methylene chloride to the top of the Snyder column. (This is important to ensure that the balls are not stuck, and the column will work properly). Attach to the KD flask.

11.9.1.3 Place the KD apparatus on a water bath (90-98^{o}C) so the tip of the concentrator tube is submerged. The water level must not reach the joint between the concentrator and the KD flask. At the proper rate of distillation, the balls will actively chatter; but the chambers should not flood.

11.9.1.4 Concentrate to 15-20 mL. If the determinative method requires a solvent exchange, add the appropriate exchange solvent to the top of the Snyder Column, and then continue the water bath concentration back down to 5-8 mL. Refer to Table 2 for details of exchange solvents and final volumes. The Snyder column may be insulated if necessary to maintain the correct rate of distillation.

Note: It is very important not to concentrate to dryness as analytes will be lost.

11.9.1.5 Remove the KD apparatus from the water bath and allow to cool for a minimum of 10 minutes. If the level of the extract is above the level of the concentrator tube joint, continue to distill the solvent as necessary. Again, allow the KD flask to cool for a minimum of 10 minutes.

11.10 Nitrogen Evaporation to Final Concentration

11.10.1 Transfer the CT to the evaporation apparatus.

11.10.2 Place the tube in a warm water bath that is at least 5°C below the boiling temperature of the solvent being evaporated and evaporate the solvent using a gentle stream of nitrogen. The nitrogen flow will form a slight depression on the surface of the solvent, but must not create splattering of the extract.

Boiling points of commonly used solvents are:

Methylene chloride	40°C
Acetone	56°C
Hexane	69°C
Acetonitrile	82°C
Toluene	111°C

Note: It is very important not to concentrate to dryness as analytes will be lost.

Note: The final concentration and volume measurement steps are critical. Use care when concentrating and make certain that the final volume measurement is accurate.

11.11 Analytical Documentation

11.11.1 Record all analytical information in the analytical logbook/logsheet which may be in an electronic format, including the analytical data from standards, blanks, LCSs, MS/MSDs, and any corrective actions or modifications to the method.

11.11.2 All standards are logged into a department standard logbook. All standards are assigned a unique number for identification. Logbooks are reviewed by the supervisor or designee.

11.11.3 Sample information and associated QC are entered into the LIMs after final technical review.

12. DATA ANALYSIS AND CALCULATIONS

Not applicable

13. METHOD PERFORMANCE

13.1. Initial Demonstration

13.1.1. Each laboratory must make an initial demonstration of capability for each individual method. This requires analysis of four QC check samples. The QC check sample is a well-characterized laboratory generated sample used to monitor method performance, which must contain all the analytes of interest. The spiking level must be equivalent to a mid-level calibration. (For certain tests, more than one set of QC check samples may be necessary in order to demonstrate capability for the full analyte list.)

13.1.2. Four aliquots of the QC check sample are analyzed using the same procedures used to analyze samples, including sample preparation.

13.1.3. Calculations and acceptance criteria for the QC check samples are given in the determinative SOPs. See SOPs NC-GC-038, NC-MS-018, NC-MS-003, and NC-GC-007 for detailed information on the determinative methods.

13.2. Training Qualification

13.2.1. The Group/Team Leader has the responsibility to ensure this procedure is performed by an analyst who has been properly trained in its use and has the required experience.

13.2.2 Method validation information (where applicable) in the form of laboratory demonstrations of capabilities is maintained for this method in the laboratory QA files

14. POLLUTION PREVENTION

14.1 It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage, and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention".

15. WASTE MANAGEMENT

15.1. All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention**."**

15.2. The following waste streams are produced when this method is carried out.

15.2.1. Extracted aqueous samples contaminated with methylene chloride. This tank is then periodically rolled to the tank room, the pH is verified, the contents are neutralized with sodium bicarbonate, the pH re-verified and the Dichloromethane waste drained into a waste drum located outside the building. The wastewater is discharged to the POTW.

15.2.2. Used sodium sulfate and glass wool or filter paper contaminated with methylene chloride/acetone or acetone/hexane from the extract drying step. These materials are disposed of in the solid waste and debris in a red container located in the Extractions Lab.

15.2.3. **Assorted flammable solvent waste from various rinses**. These wastes are put into the halogenated/non-halogenated 25 gallon solvent waste container located under the fume hood in extractions.

15.2.4. **Methylene chloride waste from various rinses:** These wastes are disposed of in the liquid-liquid separation unit.

15.2.5. **Hexane- Hexane waste:** These samples are to be disposed in the flammable waste.

15.2.6. **Waste Hexane in vials.** These vials are placed in the vial waste located in the GC prep laboratory.

15.2.7. **Waste Methylene Chloride sample vials**. These vials are placed in the vial waste located in the GC prep laboratory.

15.2.8. **Extracted solid samples contaminated with methylene chloride/acetone or acetone/hexane.** These materials are disposed of in the solid waste and debris in a red container located in the Extractions Lab.

15.2.9. Samples, standards, and all extraction materials contaminated with high levels (>50ppm) of PCBs must be segregated into their own waste stream. PCB wastes are collected in one of three waste streams, solid PCB, liquid PCB, and PCB vial waste.

15.2.10. Solvent Recovery System Waste. Methylene Chloride waste from the Solvent Recovery System is collected and disposed of in the liquid-liquid separation unit. Acetone/Methylene Chloride waste from this system is disposed of in the flammable waste containers located in the laboratory.

16. REFERENCES

16.1. References

16.1.1. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW846, 3rd Edition, Final Update III (December 1996). Sections 3500B, 3510C, 3520C, 3540C, and 3550B,

16.1.2. TestAmerica North Canton Quality Assurance Manual (QAM), current version

16.1.3. TestAmerica Corporate Environmental Health and Safety Manual, CW-E-M-001, and TestAmerica North Canton Facility Addendum and Contingency Plan, current version

16.1.4. Revision History

Historical File:		Revision 3.4: 10/16/98		Revision 4.0: 02/04/03
(formerly CORP-OP-0001NC		Revision 3.5: 04/22/99		Revision 4.1: 10/07/03
		Revision 3.6: 05/13/99		Revision 4.2: 01/30/06
		Revision 3.7: 03/20/01		Revision 0: 03/12/08 (NC-OP-032)
		Revision 3.8: 05/23/01		
		Revision 3.9: 04/22/02		

16.2. Associated SOPs and Policies, current version

16.2.1. QA Policy, QA-003

16.2.2. Glassware Washing, NC-QA-014

16.2.3. Statistical Evaluation of Data and Development of Control Charts, NC-QA-018

16.2.4. Method Detection Limits and Instrument Detection Limits, NC-QA-021 and CA-Q-S-006.

16.2.5. Supplemental Practices for DoD Project Work SOP, NC-QA-016

16.2.6. Gas Chromatographic Analysis based on Method 8000B, 8021B, 8081A, 8082, 8151A, 8015B, and 615, NC-GC-038

16.2.7. GC/MS Analysis based on Method 8270C, NC-MS-018

16.2.8. Analysis of Pesticides and PCBs by EPA Method 608, NC-GC-0007

16.2.9. GC/MS Semivolatile Organic Compounds Capillary Column Technique Based on EPA Method 625, NC-MS-003

16.2.10. Standards and Reagents, NC-QA-017

17. MISCELLANEOUS

17.1. Modifications from Reference method

17.1.1. Some surrogate spiking concentrations are modified from those recommended in SW-846, in order to make the concentrations more consistent with the calibration levels in the determinative methods.

17.1.2. Spiking levels for method 608 have been reduced by a factor of ten to bring the levels within the normal calibration range of the instrument.

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17.2. Tables

TABLE 1
Liquid /Liquid Extraction Conditions

Determinative Method	Initial Ext. pH	Secondary Ext. pH
BNA: 8270C[1]	1-2 (acid first) or 11-12 (base first)	11-12 (base first) or 1-2 (acid first)
625	11-12 (base first) or 1-2 (acid first)	1-2 (acid first) or 11-12 (base first)
Pest/PCB: 8081A, 8082 and 608	5-9	None
Hydrocarbons: 8015B	As received	None

[1] If the laboratory has validated acid only 8270 extraction for the target compound list required then the base extraction step may be omitted. The required validation consists of a four-replicate initial demonstration of capability and a method detection limit study (see Section 13). Additionally, either of the base or acid fractions of Method 8270 can be run first.

TABLE 2
Final Volumes and Exchange Solvents

Type	Exchange Solvent for Analysis	Final Volume for Analysis in mL
Semivolatiles	N/A	2.0 mL
PCB	Approximately 18 mL Hexane – water Approximately 36 mL Hexane - solid	10.0 for solids 5.0 for H2O 2.0 for H2O*
Pesticides	Approximately 18 mL Hexane	10.0 for solids 5.0 for H2O
Pesticides/TCLP	Approximately 18 mL Hexane	3.0 mL
BNA – SIM	N/A	2.0 mL - Solids & H2O
TPH	N/A	1.0

* Michigan work requires a final volume of 2 mL.

Note: Different final volumes may be necessary to meet special client reporting limit requirements.

TABLE 3

Surrogate Spiking Solutions

Analyte Group	Surrogate Spike Solution ID	Volume (mL)
BNA	100/150 ppm BNA	0.2
BNA / SIM	100/150 ppm BNA	0.2 / 0.02
BNA Waste Dilution	100/150 ppm BNA	0.5
PEST	0.2 ppm DCB/TCX	1.0
TPH	40ng Nonane (C9)	1.0
PCB	0.2 ppm DCB/TCX	1.0

TABLE 4
Matrix Spike and LCS Solutions

Analyte Group	Matrix Spike Solution ID	Volume (mL)
BNA	100 ppm BNA All-Analyte Spike and Restek Spike	0.2
	Waste Dilution	0.5
BNA / SIM	100 ppm BNA All-Analyte Spike and Restek Spike	0.2 / 0.02
PEST	Pest NPDES Spike	1.0
PEST TCLP	Pest TCLP Spike	1.0
PCB	10 ppm PCB Spike	1.0
TPH	See Spike List – Table 6	1.0

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TABLE 5
Surrogate Spike Components

Analyte Group	Compounds	Conc. (µg/mL)
BNA	2-Fluorobiphenyl	100
	Nitrobenzene-d5	100
	p-Terphenyl-d14	100
	2-Fluorophenol	150
	Phenol-d6	150
	2,4,6-Tribromophenol	150
	1,2-Dichlorobenzene-d4	100
	2-Chlorophenol-d4	150
PEST	Decachlorobiphenyl	0.2
PCB	Tetrachloro-m-xylene	0.2
TPH	Nonane (C9)	40.0

TABLE 6
Matrix Spike Components

Type	Compounds	Conc. (µg/mL)
BNA	Acenaphthene	100
	4-Chloro-3-Methylphenol	150
	2-Chlorophenol	150
	1,4-Dichlorobenzene	100
	2,4-Dinitrotoluene	100
	4-Nitrophenol	150
	N-Nitroso-Di-n-Propylamine	100
	Pentachlorophenol	150
	Phenol	150
	Pyrene	100

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Type	Compounds	Conc. (µg/mL)
BNA	1,2,4-Trichlorobenzene	100
	1,4-Dichlorobenzene	100
	2,4-Dinitrotoluene	100
	Hexachlorobenzene	100
	Hexachlorobutadiene	100
	Hexachloroethane	100
	2-Methylphenol	100
	3-Methylphenol	100
	4-Methylphenol	100
	Nitrobenzene	100
	Pentachlorophenol	100
	Pyridine	100
	2,4,5-Trichlorophenol	100
	2,4,6-Trichlorophenol	100
	Acenaphthene	100
	Acenaphthylene	100
	Anthracene	100
	Benzo(a)anthracene	100
	Benzo(b)fluoranthene	100
	Benzo(k)fluoranthene	100
	Benzo(a)pyrene	100
	Benzo(ghi)perylene	100
	Benzyl butyl phthalate	100
	Bis(2-chloroethyl)ether	100
	Bis(2-chloroethoxy)methane	100
	Bis(2-ethylhexyl)phthalate	100
	Bis(2-chloroisopropyl)ether	100
	4-Bromophenyl phenyl ether	100
	2-Chloronaphthalene	100
	4-Chlorophenyl phenyl ether	100

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Type	Compounds	Conc. (µg/mL)
BNA	Chrysene	100
	Dibenzo(a,h)anthracene	100
	Di-n-butylphthalate	100
	1,3-Dichlorobenzene	100
	1,2-Dichlorobenzene	100
	1,4-Dichlorobenzene	100
	3,3'-Dichlorobenzidine	100
	Diethyl phthalate	100
	Dimethyl phthalate	100
	2,4-Dinitrotoluene	100
	2,6-Dinitrotoluene	100
	Di-n-octylphthalate	100
	Fluoranthene	100
	Fluorene	100
	Hexachlorobenzene	100
	Hexachlorobutadiene	100
	Hexachloroethane	100
	Indeno(1,2,3-cd)pyrene	100
	Isophorone	100
	Naphthalene	100
	Nitrobenzene	100
	N-Nitrosodi-n-propylamine	100
	Phenanthrene	100
	Pyrene	100
	1,2,4-Trichlorobenzene	100
	4-Chloro-3-methylphenol	100
	2-Chlorophenol	100
	2,4-Dichlorophenol	100
	2,4-Dimethylphenol	100
	2,4-Dinitrophenol	100

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Type	Compounds	Conc. (µg/mL)
BNA	2-Methyl-4,6-dinitrophenol	100
	2-Nitrophenol	100
	4-Nitrophenol	100
	Pentachlorophenol	100
	Phenol	100
	2,4,6-Trichlorophenol	100
	Acetophenone	100
	Atrazine	100
	Caprolactum	100
	Benzaldehyde	100
	1,1′-Biphenyl	100
	Safrole	100
	1,4-Dioxane	100
	Pronamide	100
	p-Chlorobenzilate	100
	Phenacetin	100
	Ethyl methanesulfonate	100
	2-Picoline	100
	Phorate	100
	Quinoline	100

Type	Compounds	Conc. (µg/mL)
Pest TCLP	Heptachlor	0.5
	Heptachlor epoxide	0.5
	Lindane	0.5
	Endrin	0.5

Type	Compounds	Conc. (µg/mL)
	Methoxychlor	1.0
Pest NPDES/Pest	Alrin	1.0
	Alpha-BHC	1.0
	beta-BHC	1.0
	delta-BHC	1.0
	gamma-BHC (Lindane)	1.0
	4,4'-DDD	1.0
	4,4'-DDE	1.0
	4,4'-DDT	1.0
	Dieldrin	1.0
	alpha-Endosulfan	1.0
	beta-Endosulfan	1.0
	Endosulfan Sulfate	1.0
	Endrin	1.0
	Heptachlor	1.0
	Heptachlor Epoxide	1.0

Diesel Range Organics (8015B) Spike	
Compound	**Final Concentration**
n-decane	50 µg/ml
n-dodecane	50 µg/ml
n-tetradecane	50 µg/ml
n-hexadecane	50 µg/ml
n-octadecane	50 µg/ml
n-eicosane	50 µg/ml
n-docosane	50 µg/ml
n-tetracosane	50 µg/ml
n-hexacosane	50 µg/ml
n-octacosane	50 µg/ml

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Title: PREPARATION AND ANALYSIS OF MERCURY IN AQUEOUS AND SOLID SAMPLES BY COLD VAPOR ATOMIC ABSORPTION SPECTROSCOPY

[Method: MCAWW Method 245.1, SW846 Method 7470A, SW846 7471A, and 7471B]

Approvals (Signature/Date):

Technology Specialist	Date: 09/22/10	Health & Safety Coordinator	Date: 09/09/10
Quality Assurance Manager	Date: 09/22/10	Laboratory Director	Date: 09/12/10
Technical Director	Date: 09/09/10		

This SOP is a new SOP combining SOP NC-MT-011, Rev 1, dated 01/07/09 and SOP NC-MT-013 Rev 0, dated 01/07/09

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1. SCOPE AND APPLICATION

1.1. This procedure describes the preparation and analysis of mercury (Hg, CAS # 7439-97-6) by Cold Vapor Atomic Absorption Spectroscopy (CVAA) using SW846 Method 7470A, MCAWW Method 245.1 Method 7471B, and Method 7471A.

1.2. The associated LIMs method codes are BL (Method 245.1), J& (Method 7471B), O8 (Method 7470A), and O9 (Method 7471A).

1.3. CVAA analysis provides for the determination of total mercury (organic and inorganic). The combination of the oxidants and potassium permanganate has been found to give 100% recovery with both types of compounds. Detection limits, sensitivity, and optimum concentration ranges for mercury analysis will vary with the matrices, instrumentation, and volume of sample used.

1.4. Method 7470A is applicable to the preparation and analysis of mercury in ground water, aqueous samples, TCLP, and other leachates/extracts. Certain solid and sludge type wastes may also be analyzed; however, Method 7471A is usually the method of choice. All matrices require sample preparation prior to analysis.

1.5. Method 245.1 is applicable to the determination of mercury in drinking, surface and saline waters, and domestic and industrial wastes. All matrices require sample preparation prior to analysis.

1.6. Methods 7471A and 7471B are applicable to the preparation and analysis of mercury in soils, sediments, bottom deposits, wastes, wipes, biological material, and sludge-type materials. All matrices require sample preparation prior to analysis.

1.7. The TestAmerica North Canton reporting limit for mercury in aqueous matrices is 0.0002 mg/L except for TCLP or SPLP leachates for which the reporting limit is 0.002 mg/L. The TestAmerica North Canton reporting limit for mercury in solid matrices is 0.1 mg/kg.

1.8. This document accurately reflects current laboratory Standard Operating Procedures (SOP) as of the date above. All facility SOPs are maintained and updated as necessary.

2. SUMMARY OF METHOD

2.1. This SOP describes a technique for the determination of mercury in solution. The procedure is a physical method based on the absorption of radiation at 253.7 nm by mercury vapor. A representative portion of the sample is digested in sulfuric (aqueous samples), hydrochloric, and nitric acids. Organic mercury compounds are oxidized with potassium permanganate, potassium persulfate (aqueous samples), and the mercury reduced

to its elemental state with stannous chloride and aerated from solution in a closed system. The mercury vapor passes through a cell positioned in the light path of an atomic absorption spectrophotometer. Absorbance is measured as a function of mercury concentration. Concentration of the analyte in the sample is determined by comparison of the sample absorbance to the calibration curve (absorbance vs. concentration).

3. DEFINITIONS

3.1. Refer to the glossary in the TestAmerica North Canton Quality Assurance Manual (QAM), current version.

4. INTERFERENCES

4.1. Method interferences may be caused by contaminants in solvents, reagents, glassware, and other processing apparatus that lead to discrete artifacts. All of these materials must be routinely demonstrated to be free from interferences under conditions of the analysis by running laboratory method blanks as described in the Quality Control section. All glassware is cleaned per SOP NC-QA-014. Specific selection of reagents may be required to avoid introduction of contaminants.

4.2. Chemical and physical interferences may be encountered when analyzing samples using this method.

4.3. Potassium permanganate, which is used to break down organic mercury compounds, also eliminates possible interferences from sulfide. Concentrations as high as 20 mg/L of sulfide as sodium sulfide do not interfere with the recovery of inorganic mercury from reagent water.

4.4. Copper has also been reported to interfere; however, copper concentrations as high as 10 mg/L had no effect on the recovery of mercury from spiked samples.

4.5. Chlorides can cause a positive interference. Seawaters, brines, and industrial effluents high in chlorides require additional permanganate (maximum 25 mL); because during the oxidation step, chlorides are converted to free chlorine, which also absorbs radiation at 253.7 nm.

Note: Sufficient addition of permanganate is apparent when the purple color persists at least 15 minutes. Some samples may require dilution prior to digestion due to extremely high concentrations of chloride

4.6. Interference from certain volatile organic materials that absorb at this wavelength may also occur. If suspected, a preliminary run without stannous chloride can determine if this type of interference is present. While the possibility of absorption from certain organic substances

present in the sample does exist, this problem is not routinely encountered. This is mentioned only to caution the analyst of the possibility. If this condition is found to exist, the mercury concentration in the sample can be determined by subtracting the result of the sample run without the reducing reagent (stannous chloride) from that obtained with the reducing reagent.

4.7. Samples containing high concentrations of oxidizable organic materials, as evidenced by high COD levels, may not be completely oxidized by this procedure. When this occurs, the recovery of mercury will be low. The problem can be eliminated by reducing the volume of original sample used.

4.8. The most common interference is laboratory contamination, which may arise from impure reagents, dirty glassware, improper sample transfers, dirty work areas, etc. Be aware of potential sources of contamination and take appropriate measures to minimize or avoid them. Refer to Appendix B for Contamination Control Guidelines.

5. SAFETY

5.1. Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual, the Facility Addendum to the Corporate EH&S Manual, and this document.

5.2. Samples that contain high concentrations of carbonates or organic material or samples that are at elevated pH can react violently when acids are added.

5.3. The following is a list of the materials used in this method, which have a serious or significant hazard rating. NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table. A complete list of materials used in the method can be found in the Reagents and Standards section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Mercury (1,000 PPM in Reagent)	Oxidizer Corrosive Poison	0.1 g/m^3 Ceiling (Mercury Compounds)	Extremely toxic. Causes irritation to the respiratory tract. Causes irritation. Symptoms include redness and pain. May cause burns. May cause sensitization. Can be absorbed through the skin with symptoms to parallel ingestion. May affect the central nervous system. Causes irritation and burns to eyes. Symptoms include redness, pain, and blurred vision; may cause serious and permanent eye damage.

Sulfuric Acid	Corrosive Oxidizer Dehydrator Poison	1 mg/m^3-TWA	Inhalation produces damaging effects on the mucous membranes and upper respiratory tract. Symptoms may include irritation of the nose and throat, and labored breathing. Symptoms of redness, pain, and severe burn can occur. Contact can cause blurred vision, redness, pain and severe tissue burns. Can cause blindness.
Nitric Acid	Corrosive Oxidizer Poison	2 ppm-TWA 4 ppm-STEL	Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison. Inhalation of vapors can cause breathing difficulties and lead to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract. Can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Hydrochloric Acid	Corrosive Poison	5 PPM-Ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Hydroxyl-amine Hydro-chloride	Corrosive Poison	None	Extremely destructive to tissues of the mucous membranes and upper respiratory tract. Corrosive to the eyes. Irritant and possible sensitizer. May cause burns to the skin.
Potassium Persulfate	Oxidizer	None	Causes irritation to the respiratory tract. Symptoms may include coughing, shortness of breath. Causes irritation to skin and eyes. Symptoms include redness, itching, and pain. May cause dermatitis, burns, and moderate skin necrosis.
Potassium Permanganate	Oxidizer	5 mg/m^3 for Mn Compounds	Causes irritation to the respiratory tract. Symptoms may include coughing, shortness of breath. Dry crystals and concentrated solutions are caustic causing redness, pain, severe burns, brown stains in the contact area and possible hardening of outer skin layer. Diluted solutions are only mildly irritating to the skin. Eye contact with crystals (dusts) and concentrated solutions causes severe irritation, redness, and blurred vision and can cause severe damage, possibly permanent.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

5.4. Eye protection that protects against splash, laboratory coat, and appropriate gloves must be worn while samples, standards, solvents, and reagents are being handled. Cut-resistant gloves must be worn doing any other task that presents a strong possibility of getting cut. Disposable gloves that have been contaminated must be removed and discarded; other gloves must be cleaned immediately.

5.5. Exposure to chemicals must be maintained **as low as reasonably achievable.** All samples with stickers that read "Caution/Use Hood!" **must** be opened in the hood. Contact the EH&S Coordinator if this is not possible. Solvent and waste containers must be kept closed unless transfers are being made.

5.6. All work must be stopped in the event of a known or potential compromise to the health and safety of a TestAmerica North Canton associate. The situation must be reported **immediately** to the EH&S Coordinator and the Laboratory Supervisor.

5.7. Do not look directly into the beam of the Hg lamp. The UV light that these lamps radiate is harmful to the eyes.

5.8. Cylinders of compressed gas must be handled with caution in accordance with local regulations. It is recommended that, wherever possible, cylinders be located outside the laboratory, and the gas led to the instrument through approved lines.

5.9. The CVAA apparatus must be properly vented to remove potentially harmful fumes generated during sample analysis.

6. EQUIPMENT AND SUPPLIES

6.1. Temperature-controlled water bath (capable of maintaining temperature of 90- 95 °C).

6.2. Atomic Absorption Spectrophotometer equipped with:

6.2.1. Absorption cell with quartz end windows perpendicular to the longitudinal axis. Dimensions of the cell must result in sufficient sensitivity to meet the SOP defined reporting limit. The quartz windows must be maintained to provide accurate measurements. Any scratches or fingerprints can alter the absorption of UV radiation.

6.2.2. Mercury-specific hollow cathode lamp (HCL) or electrodeless discharge lamp (EDL)

6.2.3. Peristaltic pump which can deliver 1 L/min air

6.2.4. Flowmeter capable of measuring an airflow of 1 L/min

6.2.5. Recorder or printer

6.2.6. Drying device to prevent condensation in cell.

Note: Instruments designed specifically for the measurement of mercury using the cold vapor technique may be substituted for the atomic absorption spectrophotometer.

6.3. Plastic bottles – 250 mL

6.4. Nitrogen or argon gas supply, welding grade or equivalent

6.5. Calibrated automatic pipettes

6.6. Class A volumetric flasks

6.7. Top-loading balance, capable of reading up to two decimal places

6.8. Thermometer (capable of accurate readings at 95 °C)

6.9. Disposable cups or tubes

7. REAGENTS AND STANDARDS

7.1. Reagent water must be produced by a Millipore DI system or equivalent. Reagent water must be free of the analytes of interest as demonstrated through the analysis of method blanks.

7.2. Stock (10 ppm) mercury standards are purchased as custom solutions. All standards must be stored in FEP fluorocarbon or previously unused polyethylene or polypropylene bottles. Stock standard solutions must be replaced prior to the expiration date provided by the manufacturer. If no expiration date is provided, the stock solutions may be used for up to one year and must be replaced sooner if verification from an independent source indicates a problem. Additional information can be found in SOP NC-QA-017. Refer to the Standards Logbook for details on standard preparation.

7.3. Working mercury standard (0.1 ppm): Take 1 mL of the stock mercury standard (Section 7.2) and dilute to 100 mL with reagent water. The working mercury standard must be made daily and must be prepared in a matrix of 0.15% HNO_3. This acid (150 uL of concentrated HNO_3) must be added to the flask/bottle before the addition of the stock standard aliquot. Refer to the Standards Logbook for details on standard preparation.

7.4. The calibration standards must be prepared fresh daily from the working standard (Section 7.3) by transferring 0, 0.2, 0.5, 1.0, 5.0 and 10.0 mL aliquots of the working mercury standard into sample preparation bottles and proceeding as specified in Section 11.1. The LCS solution is prepared by transferring 5.0 mL of working standard (Section 7.3) into sample preparation bottles and proceeding as specified in Section 11.1. Refer to the Standards Logbook for details on standard preparation.

Note: Alternate approaches to standard preparation may be taken, and alternate volumes of standard may be prepared as long as the accuracy and final standard concentrations as detailed in Table I (Appendix A) are maintained. For example, automated mercury systems do not require 100 mL of standard and therefore smaller volumes may be generated to reduce waste generation.

7.5. The initial calibration verification standard must be made from a different stock solution than that of the calibration standards.

7.6. Refer to Table I (Appendix A) for details regarding the working standard concentrations for calibration, calibration verification and spiking solutions. All standards must be processed through the entire analytical procedure including sample preparation.

7.7. Nitric acid (HNO_3), concentrated, trace metal grade or better

7.8. Hydrochloric acid (HCl), concentrated, trace metal grade or better

7.9 Sulfuric acid (H_2SO_4), concentrated, traces metal grade or better.

7.10 Aqua Regia: Prepare immediately before use by carefully adding three volumes of concentrated HCl to one volume of concentrated HNO_3.

7.11 Stannous chloride solution: Add 50g ± 0.5g of stannous chloride and 25 mL of concentrated HCl, and bring to a final volume of 500 mL with DI water.

Note: Stannous sulfate may be used in place of stannous chloride. Prepare the stannous sulfate solution according to the recommendations provided by the instrument manufacturer.

7.12 Sodium chloride-hydroxylamine hydrochloride solution: Add 240g ± 0.5g of sodium chloride and 240g ± 0.5g of hydroxylamine hydrochloride to every 2000 mL of reagent water.

7.13 Potassium permanganate, 5% solution (w/v): Dissolve 100g of potassium permanganate for every 2000 mL of reagent water.

7.14 Potassium persulfate, 5% solution (w/v): Dissolve 100 g of potassium persulfate for every 2000 mL of reagent water.

8 SAMPLE COLLECTION, PRESERVATION, AND STORAGE

8.1 Sample holding time for mercury is 28 days from time of sample collection to the time of sample analysis.

8.2 Aqueous samples are preserved with nitric acid to a pH of <2 and may be stored in either plastic or glass. Refrigeration is not required. Preservation must be verified prior to analysis.

8.3 Soil samples and biological material do not require preservation, but must be stored at 4° C ± 2° C (and/or freezing for tissues) until the time of analysis.

9 QUALITY CONTROL

Table II (Appendix A) provides a summary of quality control requirements including type, frequency, acceptance criteria, and corrective action.

9.1 Initial Demonstration of Capability

9.2 Initial Demonstration Study - This requires the analysis of four QC check samples. The QC check sample is a well-characterized, laboratory-generated sample used to monitor method performance. The results of the initial demonstration study must be acceptable before analysis of samples may begin.

9.2.1 Four aliquots of the check sample (LCS) are prepared and analyzed using the procedures detailed in this SOP and the determinative SOPs.

9.3 Preparation Batch - A group of up to 20 samples, excluding QC Samples (LCS, Method Blank, MS, MSD), that are of the same matrix and are processed together using the same procedures and reagents. The preparation batch must contain a method blank, a LCS and a matrix spike/matrix spike duplicate. All sample setups must be initiated within a 24-hour period from the initial preparation or extraction and without interruption of the process. All samples within the batch must be treated with the same lots of reagents and the same processes. In some cases, at client request, it may be appropriate to process a matrix spike and sample duplicate in place of the MS/MSD. If clients specify specific samples for MS/MSD, the batch may contain multiple MS/MSD pairs.

9.4 Method Blank (MB) - One method blank must be processed with each preparation batch. The method blank consists of reagent water containing all reagents specific to the method that is carried through the entire analytical procedure, including preparation and analysis.

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The method blank is used to identify any system and process interferences or contamination of the analytical system that may lead to the reporting of elevated analyte concentrations or false positive data. The method blank must not contain any analyte of interest at, or above, the reporting or at, or above, 10% of the measured concentration of that analyte in associated samples, whichever is higher (sample result must be a minimum of ten times higher than the blank contamination level).

Note: For Ohio VAP projects, the result must be below the reporting limit or samples must be redigested unless the samples are non-detect.

- Repreparation and re-analysis of all samples associated with an unacceptable method blank is required when reportable concentrations are determined in the samples (see exception noted above).

- If there is no analyte greater than the RL in the samples associated with an unacceptable method blank, the data may be reported with qualifiers. **Such action must be addressed in the project narrative.**

- If the above criteria are not met and re-analysis is not possible, then the sample data must be qualified. **This anomaly must be addressed in the project narrative.**

9.5 Laboratory Control Sample (LCS) - One LCS must be processed with each preparation batch. The LCS is used to monitor the accuracy of the analytical process. Ongoing monitoring of the LCS results provides evidence that the laboratory is performing the method within acceptable accuracy and precision guidelines. The LCS must be carried through the entire analytical procedure. If the LCS is outside established control limits, the system is out of control and corrective action must occur. See Section 12 for the LCS calculation.

- In the instance where the LCS recovery is greater than the upper control limit and the sample results are less than RL, the data may be reported. Such action must be addressed in the project narrative. LCS recovery must be within 80% - 120%, or in-house control limits for 7470A. For Method 245.1, the LCS must be 85% - 115%.

- In the event an MS/MSD analysis is not possible, a Laboratory Control Sample Duplicate (LCSD) must be analyzed. The LCSD recovery is evaluated using the same control limits as the LCS.

- Corrective action must be repreparation and re-analysis of the batch unless the client agrees that other corrective action is acceptable. For Ohio VAP projects the corrective action must be repreparation and reanalysis of the batch.

9.6 Additional information on QC samples can be found in QA Policy QA-003. Ohio VAP projects must reference this SOP instead of policy QA-003 for information on QC samples.

9.7 Matrix Spike/Matrix Spike Duplicate (MS/MSD) - One MS/MSD pair must be processed for each preparation batch. A matrix spike (MS) is a field sample to which known concentrations of target analytes have been added. A matrix spike duplicate (MSD) is a second aliquot of the same sample (spiked identically as the MS) prepared and analyzed along with the sample and matrix spike. Some client-specific data quality objectives (DQOs) may require the use of sample duplicates in place of, or in addition to, MS/MSDs. The MS/MSD results are used to determine the effect of a matrix on the precision and accuracy of the analytical process. Due to the potential variability of the matrix of each sample, these results may have immediate bearing only on the specific sample spiked. Samples identified as field blanks cannot be used for MS/MSD analysis. Spiking levels are provided in Table I (Appendix I). See Section 12 for the MS/MSD and RPD calculation.

- If analyte recovery or RPD falls outside the acceptance range, the recovery of that analyte must be in control for the LCS. Until in-house control limits are established, a control limit of 70 - 130 % recovery and 20% RPD must be applied to the MS/MSD for Method 7471A. A control limit of 75 - 125 % recovery for Method 7470A, 70 – 130% for Method 245.1, and 20% RPD must be applied to the MS/MSD, until in-house limits are established. If the LCS recovery is within limits, then the laboratory operation is in control and the results may be accepted. If the recovery of the LCS is outside limits, corrective action must be taken. Corrective action must include repreparation and re-analysis of the batch. MS/MSD results which fall outside the control limits must be addressed in the narrative.

- If the native analyte concentration in the MS/MSD exceeds four times the spike level for that analyte, the recovery data are reported as “amount” MSB. The Exception Code is changed to NC. The following two footnotes will appear on the report page—“NC: The recovery and/or RPD were not calculated”, and “MSB: The recovery and RPD were not calculated, because the sample amount was greater than four times the spike amount.”

9.8 Control Limits

9.8.1 Control limits are established by the laboratory as described in SOP NC-QA-018.

9.8.2 Laboratory control limits are internally generated and updated periodically unless method specified. Control limits are easily accessible via LIMS (QC Browser program).

9.9 Method Detection Limits (MDLs) and MDL Checks

9.9.1 MDLs and MDL Checks are estab lished by the laboratory as described in SOPs NC-QA-021 and CA-Q-S-006.

9.9.2 MDLs are easily accessible via LIMS (QC Browser program)

9.10 Nonconformance and Corrective Action

9.10.1 Any deviations from QC procedures must be documented as a nonconformance with applicable cause and corrective action approved by the facility QA Manager. Procedural deviations are not allowed for Ohio VAP Projects.

10 CALIBRATION AND STANDARDIZATION

10.1 Calibration standards must be processed through the preparation procedure as described in Section 11.1.

10.2 Due to the differences in preparation protocols, separate calibration and calibration verification standards must be prepared for aqueous and solid matrices.

10.3 Calibration must be performed daily (every 24 hours) and each time the instrument is set up. The instrument calibration date and time must be included in the raw data.

10.4 Set up the instrument with the operating parameters recommended by the manufacturer. Allow the instrument to become thermally stable before beginning calibration (approximately 30 minutes of warm-up is required). Refer to the facility-specific instrument SOP and CVAA instrument manual for detailed setup and operation protocols.

10.5 Calibrate the instrument according to instrument manufacturer's instructions, using a minimum of five standards and a blank. One standard must be at, or below, the reporting limit. Analyze standards in ascending order beginning with the blank. Refer to Section 7 and Table I for additional information on preparing calibration standards and calibration levels.

10.6 The calibration curve must have a correlation coefficient of ≥ 0.995, or the instrument must be stopped and recalibrated prior to running samples. Sample results cannot be reported from a curve with an unacceptable correlation coefficient. NOTE: If any digested calibration standard does not meet SW846 criteria, all associated Ohio VAP samples must be redigested.

10.7 Initial Calibration Verification (ICV/ICB) - Calibration accuracy is verified by analyzing a second source standard (ICV). The ICV result must fall within 10% of the true value for that solution. An ICB is analyzed immediately following the ICV to monitor low level

accuracy and system cleanliness. The ICB result must fall within ± the reporting limit (RL) from zero. See Section 12 for the ICV calculation. If either the ICV or ICB fail to meet criteria, the analysis must be terminated, the problem corrected, and the instrument recalibrated (see Section 11.2.6 for required run sequence). If the cause of the ICV or ICB failure was not directly instrument-related,

the corrective action must include repreparation of the ICV, ICB, CRA, CCV, and CCB with the calibration curve. For Ohio VAP, the sample batch must be redigested.

10.8 Continuing Calibration Verification (CCV/CCB) - Calibration accuracy is monitored throughout the analytical run through the analysis of a known standard after every 10 samples. The CCV must be a mid-range standard at a concentration other than that of the ICV. For Method 245.1, the CCV must be 5% immediately following the calibration. All other CCVs for Method 245.1 must be 90-110%. The CCV result for Methods 7470A, 7471A, 7471B must fall within 20% of the true value for that solution. See Section 12 for the CCV calculation. A CCB is analyzed immediately following each CCV (see Section11.2.6 for required run sequence). The CCB result must fall within ± RL from zero. Each CCV and CCB analyzed must reflect the conditions of analysis of all associated samples. If the CCV/CCB is biased high and the sample results associated with the CCV/CCB are below the requested reporting limit, then the results can be reported. Sample results may only be reported when bracketed by valid CCV/CCB pairs. If any digested calibration standard does not meet SW846 criteria, all associated Ohio VAP samples must be redigested.

10.9 Detection Limit Standard (CRA) -To verify linearity at the reporting limit, a CRA standard is run at the beginning of each sample analysis run after the ICV/ICB. The CRA standard mercury concentration is 0.2 ug/L. It is recommended that the recovery be ± 50% of the true value, or the standard is either rerun or the problem corrected and the instrument re-calibrated. The CRA is only required when requested.

10.10 For DoD work, refer to SOP NC-QA-016 for specific details.

11 PROCEDURE

11.1 Standard and Sample Preparation Solids

11.1.1 All calibration and calibration verification standards (ICV, ICB, CCV, CCB) are processed through the digestion procedure as well as the field samples.

11.1.2 Transfer 0, 0.2, 0.5, 1.0, 5.0 and 10.0 mL aliquots of the working standard (Section 7.3) into a series of sample digestion bottles. The ICB/CCB consists of reagent water containing all reagents specific to the method that is carried through the entire analytical procedure, including preparation and analysis. For the ICV,

transfer a 2.5 ml aliquot of the working standard. The ICV working standard must be made from a source other than that used for the calibration standards. For the CCV, transfer a 5 mL aliquot of the working standard into a sample digestion bottle.

Note: Alternate volumes of standard may be prepared as long as the accuracy and final standard concentrations as detailed in Table I are maintained.

11.1.3 Add reagent water to each standard bottle to make a total volume of 10 mL. Continue preparation as described under Section 11.2 below.

11.1.4 Transfer 0.6g of a well-mixed sample into a clean sample digestion bottle. Continue preparation as described under Section 11.2.

11.2 Water Bath Protocol – Solid Samples

11.2.1 To each LCS **standard** bottle, add 5 mL of reagent water , 5 mL of aqua regia, and 5 mL of the working mercury standard (0.1 ppm) (see Section 7.3).

11.2.2 To each **sample** and Method Blank bottle, add 10 mL of reagent water and 5 mL of aqua regia.

11.2.3 Heat for two minutes in a water bath at 90 - 95 ° C.

11.2.3.1 Add 40 mL of distilled water.

11.2.3.2 Add 15 mL of potassium permanganate solution. Cover containers.

11.2.3.3 Heat for 30 minutes in the water bath at 90 - 95 °C.

11.2.3.4 Cool

11.2.3.4 Add 6 mL of sodium chloride-hydroxylamine hydrochloride solution to reduce the excess permanganate.

11.2.3.5 To each **standard** and sample bottles, add 50 mL of reagent water.

11.2.3.6 Continue as described under Section 11.5.

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11.3 Standard and Sample Preparation Waters

11.3.1 All calibration and calibration verification standards (ICV, ICB, CCV, CCB) are processed through the digestion procedure as well as the field samples. Transfer 0, 0.2, 0.5, 1.0, 5.0 and 10.0 mL aliquots of the working standard (Section 7.3) into a series of 100 ml class A volumetrics, then dilute to volume. For the ICV, use a 2.5 ml aliquot of the working standard. The ICV working standard must be made from a source other than that used for the calibration standards. For the CCV, transfer a 5 mL aliquot of the working standard into a Class A volumetric, then dilute to volume. The Method Blank consists of 100 mL of reagent water.

Note: Alternate volumes of standard may be prepared as long as the accuracy and final standard concentrations as detailed in Table I are maintained.

11.3.2 Transfer 100 mL of well-mixed sample or standard to a clean sample digestion bottle. Continue preparation as described under Section 11.4.

Note: Reduced sample volumes can be used as long as a representative sample can be obtained and the reagent levels are adjusted to maintain the same sample to reagent ratio. All samples and standards must be processed similarly.

Note: Spiking is done before the addition of acids or reagents.

11.4 Water Bath Protocol – Water Samples

11.4.2 Add 5 mL of concentrated H_2SO_4 and 2.5 mL of concentrated HNO_3.

11.4.3 Add 15 mL of potassium permanganate solution. For samples high in organic materials or chlorides, additional permanganate may be added. Shake and add additional portions of permanganate solution until a purple color persists for at least 15 minutes. If after the addition of up to 25-mL additional permanganate the color does not persist, sample dilution prior to re-analysis may be required.

Note: The sample dilution resultant from the addition of more than the original aliquot of permanganate solution must be compensated for in the final calculation.

11.4.4 Add 8 mL of potassium persulfate solution, cover, and heat for two hours in a water bath at 90 - 95 °C.

11.4.5 Cool samples.

11.4.6 Add 6 mL of sodium chloride-hydroxylamine hydrochloride solution to reduce the excess permanganate.

11.5 Sample Analysis

11.5.1 Automated determination. Refer to Appendix C for instrument setup and operation.

11.5.2 Perform a linear regression analysis of the calibration standards by plotting maximum response of the standards vs. concentration of mercury. Determine the mercury concentration in the samples from the linear regression fit of the calibration curve. The calibration acceptance criteria is listed in Section 10.6. Calibration using computer or calculation based regression curve fitting techniques on concentration/response data is acceptable.

11.5.3 All measurements must fall within the defined calibration range to be valid. Dilute and re-analyze all samples for analytes that exceed the highest calibration standard.

11.5.4 The following analytical sequence is consistent with Methods 7470A, 245.1, and 7471A.

Instrument Calibration
ICV
ICB
CRA if required
Maximum 10 samples
CCV
CCB
Repeat sequence of 10 samples between CCV/CCB pairs as required to complete run
CCV
CCB

Refer to Quality Control Section 9.0 and Table II (Appendix A) for quality control criteria.

Note: Samples include the method blank, LCS, MS, MSD, duplicate, field samples and sample dilutions.

11.5.5 To facilitate the early identification of QC failures and samples requiring rerun, it is strongly recommended that sample data be reviewed periodically throughout the run.

11.5.6 Guidelines are provided in the appendices on procedures to minimize contamination of samples and standards, preventive maintenance, and troubleshooting.

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11.6 Analytical Documentation

11.6.2 Record all analytical information in the analytical logbook/logsheet, which may be in electronic format, including the analytical data from standards, blanks, LCSs, MS/MSDs, and any corrective actions or modifications to the method.

11.6.3 All standards and reagents are logged into a department standard logbook. All standards are assigned a unique number for identification. Logbooks are reviewed by the supervisor or designee.

11.6.4 Documentation such as all associated instrument printouts (final runs, screens, reruns, QC samples, etc.) and daily calibration data corresponding to all final runs is available for each data file.

11.6.5 Sample results and associated QC are entered into LIMs after final technical review.

11.6.6 One-time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, chemistry, sample size, or other parameters. Any variation in procedure must be completely documented using a Nonconformance Memo. The Nonconformance Memo must be filed in the project file.

11.6.7 Any unauthorized deviations from this procedure must also be documented as a nonconformance with a cause and corrective action described. Procedural deviations are not allowed for Ohio VAP projects.

12. DATA ANALYSIS AND CALCULATIONS

12.1 ICV percent recoveries are calculated according to the equation:

$$\%R = 100\left(\frac{Found(ICV)}{True(ICV)}\right)$$

12.2 CCV percent recoveries are calculated according to the equation:

$$\%R = 100\left(\frac{Found(CCV)}{True(CCV)}\right)$$

12.3 Matrix spike recoveries are calculated according to the following equation:

$$\%R = 100\left(\frac{SSR - SR}{SA}\right)$$

Where:

SSR = Spike Sample Result
SR = Sample Result
SA = Spike Added

12.4 The relative percent difference (RPD) of matrix spike/matrix spike duplicates or sample duplicates are calculated according to the following equations:

$$RPD = 100\left[\frac{|MSD - MS|}{\left(\frac{MSD + MS}{2}\right)}\right]$$

Where:

MS = determined spiked sample concentration
MSD = determined matrix spike duplicate concentration

$$RPD = 100\left[\frac{|DU1 - DU2|}{\left(\frac{DU1 + DU2}{2}\right)}\right]$$

Where:

DU1 = Sample result
DU2 = Sample duplicate result

12.5 The final concentration determined in solid samples when reported on a dry weight basis is calculated as follows:

$$mg/kg, dry\ weight = (C \times V \times D)/(W \times S)$$

Where:

C = Concentration (ug/L) from instrument readout
V = Volume of digestate (L)
D = Instrument dilution factor
W = Weight in g of wet sample digested
S = Percent solids/100

Note: A Percent Solids determination must be performed on a separate aliquot when dry weight concentrations are to be reported. If the results are to be reported on a wet weight basis, the "S" factor must be omitted from the above equation.

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12.6 The final concentration for an aqueous sample is calculated as follows:

$$mg/L = C \times D$$

Where:

C = Concentration (mg/L) from instrument readout
D = Instrument dilution factor

12.7 The LCS percent recovery is calculated according to the following equation:

$$\%R = 100\left(\frac{Found(LCS)}{True(LCS)}\right)$$

12.8 Appropriate factors must be applied to sample values if dilutions are performed.

12.9 Additional equations and calculations are listed in the following SOPs: Calibration Curves (General), CA-Q-S-005, and Selection of Calibration Points, CA-T-P-002.

13. METHOD PERFORMANCE

13.1 Each laboratory must have initial demonstration of performance data on file for each analyte of interest as described in Section 9.1.

13.2 Training Qualification

13.2.1 The Group/Team Leader has the responsibility to ensure this procedure is performed by an associate who has been properly trained in its use and has the required experience.

14 POLLUTION PREVENTION

14.1 It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage, and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention".

15 WASTE MANAGEMENT

15.1 All waste must be disposed of in accordance with Federal, State, and Local laws and regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method

and the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention."

15.2 Waste Streams Produced by this Method

15.2.1 The following waste streams are generated by this method.

15.2.1.1 Acid Waste. This waste disposed of in the designated container labeled "Acid Waste".

15.2.1.2 Acid waste-aqueous waste generated by the analysis. Samples are disposed of in the acid waste drum located in the Metals lab. The contents of the drum are neutralized and released to the POTW.

16. REFERENCES

16.1 References

16.1.1 Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW846, 3rd Edition, Final Update II, Revision I, September 1994, Method 7470A (Mercury)

16.1.2 "Methods for the Chemical Analysis of Water and Wastes", Rev. 3.0 (1994)

16.1.3 Test Methods for Evaluating Solid Waste , Physical/Chemical Methods, SW-846, 3rd Edition, Final Update II, Revision I, September 1994, Method 7471A (Mercury)

16.1.4 Test Methods for Evaluating Solid Waste , Physical/Chemical Methods, SW-846, 3rd Edition, Revision 2, January 1998, Method 7471B (Mercury)

16.1.5 TestAmerica North Canton Quality Assurance Manual (QAM), current version

16.1.6 TestAmerica Corporate Environmental Health and Safety Manual, CW-E-M-001, and TestAmerica North Canton Facility Addendum and Contingency Plan, current version

16.1.7 Corporate Quality Management Plan (CQMP), current version

16.1.8 Revision History

Historical File:		Revision 1.1: 04/17/97		(NC-MT-011) Revision 0: 12/12/07
(formerly CORP-MT-0007NC)		Revision 2.2: 02/06/01		(NC-MT-011 Rev 1 & NC-MT-013 Rev 0): 01/07/09
		Revision 2.3: 05/15/01		
		Revision 2.4: 10/28/02		
		Revision 2.5: 11/22/04		

16.2 Associated SOPs and Policies, current version

16.2.1. QA Policy, QA-003

16.2.2. Glassware Washing, NC-QA-014

16.2.3. Statistical Evaluation of Data and Development of Control Charts, NC-QA-018

16.2.4. Method Detection Limits and Instrument Detection Limits, NC-QA-021 and CA-Q-S-006

16.2.5. Supplemental Practices for DoD Project Work, NC-QA-016

16.2.6. Standards and Reagents, NC-QA-017

16.2.7. Calibration Curves (General), CA-Q-S-005

16.2.8 Section of Calibration Points, CA-T-P-002

16.2.9 Subsampling, NC-IP-001

17. MISCELLANEOUS (TABLES, APPENDICES, ETC.)

17.1 Modifications/Interpretations from Reference Method

17.1.1 Modifications from Method 7471A

17.1.1.1 Chapter 1 of SW846 specify the use of reagent water with a purity equivalent to ASTM Type II water. This SOP specifies the use of a Millipore DI system or equivalent to produce reagent water. This SOP requires that reagent water must be free of the analytes of interest as demonstrated through the analysis of method blanks.

17.1.1.2 Chapter 1 of SW-846 states that the method blank must not contain any analyte of interest at or above the MDL. This SOP states that the method blank must not contain any analyte of interest at or above the reporting limit.

17.1.2 Modifications from both Methods 7470A and 245.1

17.1.2.1 The 200 series methods and Chapter 1 of SW846 specify the use of reagent water with a purity equivalent to ASTM Type II water. This SOP specifies the use of a Millipore DI system or equivalent to produce reagent water. This SOP requires that reagent water must be free of the analytes of interest as demonstrated through the analysis of method blanks.

17.1.2.2 This SOP allows for the use of reduced sample volumes to decrease waste generation. Reagent levels are adjusted to maintain the same ratios as stated in the source methods. According to a letter from Robert Booth of EPA EMSL-Cinn to David Payne of EPA Region V, "Reduction in sample size and appropriate corresponding reduction in sample volume is not considered a significant change in the methodology."

17.1.3 Modifications from Method 7470A

17.1.3.1 Chapter 1 of SW-846 states that the method blank must not contain any analyte of interest at or above the MDL. This SOP states that the method blank must not contain any analyte of interest at or above the reporting limit if the samples associated with the method blank are equal to or above the reporting limit.

17.1.4 Modifications from Method 245.1

17.1.4.1 Method 245.1 states that standards are not heated. TestAmerica North Canton prepares heated standards for this method.

17.1.4.2 Stannous Chloride is prepared in hydrochloric acid, instead of sulfuric acid, per instrument manufacturer recommendations.

17.1.4.3 Section 9.3.4 of the method states that the CCB must be less than the MDL. The laboratory uses the criteria that the CCB result must fall within ± RL from zero.

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APPENDIX A - TABLES

TABLE I . MERCURY REPORTING LIMITS, CALIBRATION STANDARD, QC STANDARD AND SPIKING LEVELS

Soil RL (mg/kg)	0.1
Standard Aqueous RL	0.0002
TCLP RL	0.002
Std 0 (mg/L)	0
Std 1/CRA (mg/L)	0.0002
Std 2 (mg/L)	0.0005
Std 3 (mg/L)	0.001
Std 4 (mg/L)	0.005
Std 5 (mg/L)	0.010
ICV (mg/L)	0.0025
CCV/LCS/LCSD (mg/L)	0.005
MS (mg/L)	0.001
TCLP MS	0.005

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TABLE II. SUMMARY OF QUALITY CONTROL REQUIREMENTS

QC Parameter	Frequency	Acceptance Criteria	Corrective Action
ICAL	Daily (every 24 hours)	Curve consists of 5 standards and a blank. Correlation Coefficient of 0.995 or higher and passing ICV/ICB pair.	Recalibrate. If any digested calibration standard does not meet SW846 criteria, all associated Ohio VAP samples must be redigested.
ICV	Beginning of every analytical run	90 - 110 % recovery	Terminate analysis, correct the problem, recalibrate, or reprep with calibration curve (see Section 10). Redigestion of samples is required for OVAP.
ICB	Beginning of every analytical run, immediately following the ICV	The result must be within ± RL from zero	Terminate analysis, correct the problem, recalibrate, or reprep with calibration curve (see Section 10). Redigestion of the samples is also required for OVAP.
CRA	Beginning of every analytical run following the ICB and prior to sample analyses at or near the end of a run required only when requested	Recommended recovery of 50%-150%	Rerun to verify; or correct problem and recalibrate or reprep with the calibration curve (See Sec. 10). CRA only required when requested.
CCV	Every 10 samples and at the end of the run	80 - 120 % recovery for solid samples. Recovery for Method 245.1 = 95-105% following a calibration. All other CCVs for 245.1 are 90-110%. Recovery for Method 7470A, 7471A and 7471B = 80-120% following a calibration. If the CCV is biased high and the samples are < RL the results are acceptable.	Terminate analysis, correct the problem, recalibrate, and rerun all samples not bracketed by acceptable CCV or reprep with calibration curve. If CCV is biased high and samples are ND, results can be reported (see Section 10). If any digested standard does not meet SW846 criteria, all associated Ohio VAP samples must be redigested

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TABLE II. SUMMARY OF QUALITY CONTROL REQUIREMENTS

QC Parameter	Frequency	Acceptance Criteria	Corrective Action
CCB	Immediately following each CCV	The result must be within ± RL from zero	Terminate analysis, correct the problem, recalibrate, and rerun all samples not bracketed by acceptable CCB or reprep with calibration curve. Redigestion of the samples is also required for OVAP. If CCB is biased high and samples are ND, results can be reported (see Section 10).
Method Blank	One per sample preparation batch of up to 20 samples	The result must be less than or equal to the RL Sample results greater than 20x the blank concentration are acceptable Samples for which the contaminant is < RL do not require redigestion (see Section 9.4)	Redigest and reanalyze samples. Note exceptions under criteria section. See Section 9.4 for additional requirements.
Laboratory Control Sample/Laboratory Control Sample Duplicate(LCS/LCSD)	One per sample preparation batch of up to 20 samples	Aqueous LCS/LCSD must be within 80 - 120% recovery or in-house control limits For Method 245.1, the LCS must be 85 – 115%.	Terminate analysis, correct the problem, redigest, and re-analyze all samples associated with the LCS (see Section 9.5).
Matrix Spike	One per sample preparation batch of up to 20 samples	75 - 125 % recovery or in-house control limits for 7470A. For Method 245.1, recovery limits are 70% - 130%. If the MS/MSD is out for an analyte, it must be in control in the LCS	In the absence of client-specific requirements, flag the data. No flag required if the sample level is > 4 times the spike added (see Section 10).
Matrix Spike Duplicate	See Matrix Spike	75 - 125 % recovery or in-house control limits for 7470A. For Method 245.1, recovery limits are 70% - 130%. RPD ≤ 20% (see MS)	See Corrective Action for Matrix Spike

APPENDIX B - CONTAMINATION CONTROL GUIDELINES

The following procedures are strongly recommended to prevent contamination:

All glassware should be washed with detergent and tap water and rinsed with 1:1 nitric acid followed by deionized water.

Proper laboratory housekeeping is essential in the reduction of contamination in the metals laboratory. All work areas should be kept scrupulously clean.

Powdered Gloves should not be used in the metals laboratory since the powder contains zinc, as well as other metallic analytes. Glassware should be periodically checked for cracks and etches and discarded if found. Etched glassware can cause cross contamination of any metallic analytes.

Autosampler trays should be covered to reduce the possibility of contamination. Trace levels of elements being analyzed in the samples can be easily contaminated by dust particles in the laboratory.

The following are helpful hints in the identification of the source of contaminants:

Reagents or standards can contain contaminants or be contaminated with the improper use of a pipette.

Improper cleaning of glassware can cause contamination.

Separate glassware if an unusually high sample is analyzed and soak with sulfuric acid prior to routine cleaning.

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APPENDIX C – INSTRUMENT SETUP

Hg Analysis (Leeman PS200II)
System Initialization and Warmup

To Set Up Instrument for Analysis

1. F1 Menu
2. **A**utosampler
    A. **R**ack Entry
    B. **E**dit (ex. Rack 1), Enter
    C. Cup **I**D - Enter (clears sample #'s)
    E. Press Insert Key and move cursor with arrows to cup ID and begin typing labels.
3. Press F2 Macro key and type in – Hg
    A. Enter folder name - ex., HG0306, Enter. If folder does not exist, type Y - Enter.
    B. Type in: "Rack 1", "Rack 2" etc., Enter.
    C. Type in: FROM CUP TO CUP
        Ex. = 1 30

Do the same for Position 2 if needed. If not needed, you must press "Enter" three times to begin analysis.

Hg Analysis (HYDRA AA)

Instrument Instruction

H4 Analysis

There are 3 separate screens to use--WinHgRunner, WinHgDatabase, and Rack Editor

(Turn on Hg lamp prior to analysis)

1. To Set your Protocol and Dataset

   WinHgDatabase – Select a previous protocol, then Save Protocol. Type in file name (ex.: HG40101), then select the (RN) key. This will take you to the WinHgRunner screen.

   WinHgRunner – File, New, type in dataset name (ex.: 0101A). Type in batch name (ex.: Water or Solid). Go back to WinHgDatabase to locate the new protocol.

2. Typing Labels

   Rack Editor - File, New (pick 44 rack). Type in labels under sample ID, and Save As (ex.: 0101A).

3. Activate Gas and Pump

   WinHgRunner – under Control tab, turn on Gas and pump, and pour calibration standards.

4. To Calibrate Curve

   WinHgRunner – under Standard tab, select S1 S2 S3 S4 S5 S6 Rep1. To begin analyzing, select Stnd Auto tab.

5. To Check Calibration Curve

   WinHgDatabase – under Cal Curve tab linear range >0.995, then accept curve.

6. Verification Standards

   WinHgRunner – under the Standard tab, select C1 (ICV) C2 (ICB) C3 (CRA). To begin analysis, select Ck Std Auto tab.

7. Checking Verification Standards:

   WinHgRunner – Select the Report Tab to review results.

8. To Begin Analyzing Sample with CCV and CCB:

   WinHgRunner – under Standard tab, select C4 (CCB) and C5 (CCV).

   WinHgRunner – under Sample tab, type in rack name, start cup, end cup, cups per rack (44). To start analysis, select the Run Auto tab.

9. View Results

   WinHgRunner – Select the Report tab.

PRINTING REPORTS

1. To Print Cal Curve

   WinHgDatabase – under Cal Curve tab**,** select Print Cal to print curve.

2. To Print Report

   WinHgDatabase – under Report tab under Format, turn on Report. Then select Generate to print.

3. To Transfer Run

   WinHgDatabase – under Report tab under Format, turn on PRN file and type in file (ex. H:/40101A). Then select Generate to transfer run.

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Title: INDUCTIVELY COUPLED PLASMA – ATOMIC EMISSION SPECTROSCOPY, SPECTROMETRIC METHOD FOR TRACE ELEMENT ANALYSES

[Method: SW846 Methods 6010A,6010B and EPA Method 200.7]

Approvals (Signature/Date):

Karen R. Counts 1/5/09
Technology Specialist Date

1/06/09
Health & Safety Coordinator Date

Dorothy J. Lusson 1/5/09
Quality Assurance Manager Date

1/5/09
Laboratory Director Date

1/5/09
Technical Director Date

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1. SCOPE AND APPLICATION

1.1. This procedure describes the analysis of trace elements including metals in solution by Inductively Coupled Plasma -Atomic Emission Spectroscopy (ICP-AES) using SW-846 Methods 6010A, 6010B and EPA Method 200.7. Tables I and IA of Appendix A lists the elements appropriate for analysis by Methods 6010A, 6010B and 200.7 and the associated reporting limit.. Additional elements may be analyzed under Methods 6010A, 6010B and 200.7 provided that the method performance criteria presented in Section 13.0 are met.

1.2. ICP analysis provides for the determination of metal concentrations over several orders of magnitude. Detection limits, sensitivity, and optimum concentration ranges of the metals will vary with the matrices and instrumentation used.

1.3. Methods 6010A and 6010B are applicable to the determination of dissolved, suspended, total recoverable, and total elements in ground water, aqueous samples, soils, sludges, wastes, sediments, biological, and TCLP, EP, and other leachates/extracts. All matrices require digestion prior to analysis. Silver concentrations must be below 2.0 mg/L in aqueous samples and 100 mg/kg in solid matrix samples. Precipitation may occur in samples where silver concentrations exceed these levels and lead to the generation of erroneous data.

1.4. Method 200.7 is applicable to the determination of dissolved, suspended, total recoverable, and total elements in water, waste water, and solid wastes. All matrices require digestion prior to analysis. Silver concentrations must be below 0.1 mg/L in aqueous samples.

2. SUMMARY OF METHOD

2.1. This method describes a technique for the determination of multi elements in solution using sequential or simultaneous optical systems and axial or radial viewing of the plasma. The basis of the method is the measurement of atomic emission by an optical spectroscopic technique. Samples are nebulized and the aerosol that is produced is transported to the plasma torch where excitation occurs. Characteristic atomic-line emission spectra are produced by radio frequency inductively-coupled plasma (ICP). The spectra are dispersed by a grating spectrometer and the intensities of the emission lines are monitored by photomultiplier tubes. The photocurrents from the photomultiplier tubes are processed and controlled by a computer system. A background correction technique is required to compensate for variable background contribution to the determination of trace elements. Background must be measured adjacent to analyte lines during analysis. The position selected for the background intensity measurement, on either or both sides of the analytical line, will be determined by the complexity of the spectrum adjacent to the analyte line. The position used must be free of spectral interferences and reflect the same change in

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background intensity as occurs at the analyte wavelength measured. Background correction is not required in cases of line broadening where a background correction measurement would actually degrade the analytical result. The possibility of additional interferences must also be recognized, and appropriate actions taken. Alternatively, multivariate calibration methods may be chosen for which point selection for background correction is superfluous since whole spectral regions are processed.

2.1. Refer toNC-IP-010, Acid Digestion of Soils, SW846 Method 3050B, andNC-IP-011, Acid Digestion of Aqueous Samples by SW846 and MCAWW 200 Series Methods for details on sample preparation methods.

3. DEFINITIONS

3.1. Dissolved Metals: Those elements which pass through a 0.45 um membrane. (Sample is acidified after filtration).

3.2. Total Metals: The concentration determined on an unfiltered sample following vigorous digestion.

3.3. Total Recoverable Metals: The concentration determined on an unfiltered sample following treatment with hot, dilute mineral acid.

3.4. Refer to the glossary in the TestAmerica North Canton Quality Assurance Manual (QAM), current version for additional definitions. Refer to Appendix B for a cross reference of method definitions.

4. INTERFERENCES

4.1. Spectral, physical and chemical interference effects may contribute to inaccuracies in the determinations of trace elements by ICP. Spectral interferences are caused by:

- Overlap of a spectral line from another element.
- Unresolved overlap of molecular band spectra.
- Background contribution from continuous or recombination phenomena.
- Stray light from the line emission of high concentration elements.

4.1.1. A background correction technique is required to compensate for variable background contribution to the determination of trace elements. Background correction is not required in cases where a background corrective measurement would actually degrade the analytical result.

4.1.2. Inter-element correction factors (IECs) are necessary to compensate for spectral overlap. Inter-element interferences occur when elements in the

sample emit radiation at wavelengths so close to that of the analyte that they contribute significant intensity to the analyte channel. If such conditions exist, the intensity contributed by the matrix elements will cause an excessively high (or sometimes low) concentration to be reported for the analyte. Inter-element corrections IECs must be applied to the analyte to remove the effects of these unwanted emissions.

4.1.3. Physical interferences are generally considered to be effects associated with sample transport, nebulization, and conversion within the plasma. These interferences may result in differences between instrument responses for the sample and the calibration standards. Physical interferences may occur in the transfer of solution to the nebulizer (e.g., viscosity effects) at the point of aerosol formation and transport to the plasma (e.g., surface tension) or during excitation and ionization processes within the plasma itself. Changes in viscosity and surface tension can cause significant inaccuracies, especially in samples containing high dissolved solids or high acid concentrations. If physical interferences are present, dilution of the sample, use of a peristaltic pump, mass flow controller, use of an internal standard, and/or use of a high solids nebulizer can reduce the effect. Chemical interferences are characterized by molecular compound formation, ionization effects, and solute vaporization effects. Normally these effects are not significant with the ICP technique, but if observed can be minimized by buffering the sample, matrix matching, or standard addition procedures.

5. SAFETY

5.1. Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual and this document.

5.2. Eye protection that protects against splash, laboratory coat, and appropriate gloves must be worn while samples, standards, solvents, and reagents are being handled. Disposable gloves that have been contaminated must be removed and discarded; other gloves must be cleaned immediately.

5.3. The following is a list of the materials used in this method, which have a serious or significant hazard rating. **NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the Reagents and Standards section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure

Nitric Acid	Corrosive Oxidizer Poison	2 ppm-TWA 4-ppm STEL	Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison. Inhalation of vapors can cause breathing difficulties and lead to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract. Can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Hydrochloric Acid	Corrosive Poison	5 ppm-Ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
1 – Always add acid to water to prevent violent reactions			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

5.3.1. The plasma emits strong UV light and is harmful to vision. **NOTE**: **AVOID looking directly at the plasma**.

5.3.2. The RF generator produces strong radio frequency waves, most of which are unshielded. People with pacemakers must not go near the instrument while in operation.

5.4. Exposure to chemicals must be maintained **as low as reasonably achievable.** All samples with stickers that read "Caution/Use Hood!" **must** be opened in the hood. Contact the EH&S Coordinator if this is not possible. Metals digestates can be processed outside of a fume hood. Solvent and waste containers must be kept closed unless transfers are being made.

5.5. All work must be stopped in the event of a known or potential compromise to the health and safety of a TestAmerica North Canton associate. The situation must be reported **immediately** to the EH&S Coordinator and the Laboratory Supervisor.

6. EQUIPMENT AND SUPPLIES

6.1. Inductively Coupled Plasma Atomic Emission Spectrometer equipped with autosampler and background correction.

6.2. Radio Frequency Generator

6.3. Argon gas supply, welding grade or equivalent

6.4. Coolflow or appropriate water cooling device

6.5. Peristaltic Pump

6.6. Calibrated automatic pipettes or Class A glass volumetric pipettes – ranging from 5 µL → 10 ml

6.7. Class A volumetric flasks – range from 50 ml → 2000 ml

6.8. Autosampler tubes – range from 8 ml → 14 ml

7. REAGENTS AND STANDARDS

7.1 Intermediate standards are purchased as custom multi-element mixes or as single-element solutions. All standards must be stored in FEP fluorocarbon or unused polyethylene or polypropylene bottles. Intermediate standard solutions must be replaced prior to the expiration date provided by the manufacturer. If no expiration date is provided, the intermediate solutions may be used for up to one year and must be replaced sooner if verification from an independent source indicates a problem. Expiration dates can be extended provided that the acceptance criteria described in laboratory-specific SOPs are met. Additional information can be found in SOP NC-QA-017.

7.2 Working calibration, calibration verification solutions, and internal standard solutions must be prepared in a matrix of 5% hydrochloric and 5% nitric acids. Refer to Tables II, III, IV and V (Appendix A) for details regarding the working standard concentrations for calibration, calibration verification, interference correction, and spiking solutions. Refer to the laboratory Standard Logbook or Reagent Logbook for details on standard or reagent preparation.

7.3 Concentrated nitric acid (HNO_3), trace metal grade or better.

7.4 Concentrated hydrochloric acid (HCl), trace metal grade or better.

7.5 Reagent water must be produced by a Millipore DI system or equivalent. Reagent water must be free of the analytes of interest as demonstrated through the analysis of method blanks.

8. SAMPLE COLLECTION, PRESERVATION AND STORAGE

8.1. Sample holding times for metals are six months from time of collection to the time of analysis.

8.2. Aqueous samples are preserved with nitric acid to a pH of <2 and may be stored in

either plastic or glass. If boron is to be determined, plastic containers are preferred. Refrigeration is not required. Preservation must be verified prior to analysis.

8.3. Soil samples do not require preservation, but must be stored at 4°C ± 2° until the time of preparation.

9. QUALITY CONTROL

Table VI (Appendix A) provides a summary of quality control requirements including type, frequency, acceptance criteria, and corrective action.

9.1. Initial Demonstration of Capability

9.1.1. Prior to analysis of any analyte using Methods 200.7, 6010A, or 6010B, the following requirements must be met.

9.1.2. Instrument Detection Limit (IDL) - The IDL for each analyte must be determined for each analyte wavelength used for each instrument. The IDL must be determined annually for client-specific projects. For DoD work, refer to SOP NC-QA-016. If the instrument is adjusted in any way that may affect the IDL, the IDL for that instrument must be redetermined. The IDL will be determined by multiplying by 3, the standard deviation obtained from the analysis of a blank solution, with seven consecutive measurements. Each measurement must be performed as though it were a separate analytical sample (i.e., each measurement must be followed by a rinse and/or any other procedure performed between the analysis of separate samples).

9.1.3. Method Detection Limit (MDL) - An MDL must be determined for each analyte prior to the analysis of any client samples. Refer to TestAmerica North Canton SOP NC-QA-021 and CA-Q-S-006 for details on MDL analysis and criteria.

9.1.4. Linear Range Verification (LR) - The linear range must be verified every six months for each analyte wavelength used on each instrument. The linear range is the concentration above which results cannot be reported without dilution of the sample. The standards used to verify the linear range limit must be analyzed during a routine analytical run, and must read within 10% of the expected value.

For the **initial** determination of the upper limit of the linear dynamic range (LDR) for each wavelength, determine the signal responses from a minimum of three to five different concentration standards across the estimated range. One standard must be near the upper limit of the estimated range. The concentration measured at the LDR must be no more than 10% less than the expected level extrapolated from lower standards. If the instrument is adjusted in any way that may affect the LRs, new dynamic ranges must be determined. The LR data must be documented and kept on file.

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9.1.5. Background Correction Points - To determine the appropriate location for off-line background correction when establishing methods, the user must scan the area on either side adjacent to the wavelength and record the apparent emission intensity from all other method analytes. This spectral information must be documented and kept on file. The location selected for background correction must be either free of off-line inter-element spectral interference, or a computer routine must be used for automatic correction on all determinations. Tests to determine spectral interference must be done using analyte concentrations that will adequately describe the interference. Background correction points must be set prior to determining IECs. Refer to the ICP instrument manual for specific procedures to be used in setting background correction points.

9.1.6. Inter-element Corrections (IECs) - ICP inter-element correction factors must be determined prior to the analysis of samples and every six months thereafter. If the instrument is adjusted in any way that may affect the IECs, the IECs must be redetermined. When initially determining IECs for an instrument, wavelength scans must be performed to ensure that solutions in use are free from contaminants. If an IEC varies significantly from the previously determined IEC, then the possibility of contamination must be investigated. The purity of the IEC check solution can be verified by using a standard from a second source or an alternate method (i.e., ICP-MS). Published wavelength tables (e.g., MIT tables, Inductively Coupled Plasma-Atomic Spectroscopy: Prominent Lines) can also be consulted to evaluate the validity of the IECs. Refer to the instrument manufacturer's recommendations for specific procedures to be used in setting IECs. An IEC must be established to compensate for any interelement interference which results in a false analyte signal greater than ± the RL as defined in Tables I or IA. For elements with a reporting limit of 10 ug/L or less, the signal must be ± two times the RL. To determine IECs, run a single element standard at the established linear range. To calculate an IEC, divide the observed concentration of the analyte by the actual concentration of the "interfering element."

Note: Trace ICP IECs are more sensitive to small changes in the plasma and instrument setup conditions. Adjustments in the IECs will be required on a more frequent basis for the Trace as reflected by the ICSA response. Additional spectral interference is present from easily ionizable elements such as potassium and sodium in axial viewing instruments.

9.1.7. Rinse Time Determination - Rinse times must be determined upon initial set-up of an ICP instrument. To determine the appropriate rinse time for a particular ICP system, the linear range verification standard (see Section 9.1.4) must be aspirated as a regular sample followed by the analysis of a series of rinse blanks. The length of time required to reduce the analyte

signals to < RL will define the rinse time for a particular ICP system. For some analytes, it may be impractical to set the rinse time based on the linear range standard result (i.e., analyte not typically detected in environmental samples at that level and an excessive rinse time would be required at the linear range level). Until the required rinse time is established, the method recommends a rinse period of at least 60 seconds between samples and standards. If a memory effect is suspected, the sample must be re-analyzed after a rinse period of sufficient length. Rinse time studies can be conducted at additional concentration levels. These additional studies must be documented and kept on file, if a concentration other than the linear range level is used to set the rinse time. The concentration levels used to establish the rinse time must be taken into consideration when reviewing the data.

9.2. Method Blank (MB) - One method blank must be processed with each preparation batch. The method blank consists of reagent water containing all reagents specific to the method that is carried through the entire analytical procedure, including preparation and analysis. The method blank is used to identify any system and process interferences or contamination of the analytical system that may lead to the reporting of elevated analyte concentrations or false positive data. The method blank must not contain any analyte of interest at or above the reporting limit (exception: common laboratory contaminants, see below) or at or above 5% of the measured concentration of that analyte in associated samples, whichever is higher (sample result must be a minimum of 20 times higher than the blank contamination level). For Ohio VAP projects, all analytes must be less than the reporting limit unless the associated samples are ND.

- If the analyte is a common laboratory contaminant (copper, iron, lead, or zinc), the data may be reported with qualifiers if the concentration of the analyte in the method blank is less than two times the RL. Such action must be addressed in the project narrative.

- Repreparation and re-analysis of all samples associated with an unacceptable method blank is required when reportable concentrations are determined in the samples (see exception noted above).

- If there is no analyte greater than the RL in the samples associated with an unacceptable method blank, the data may be reported with qualifiers. Such action must be addressed in the project narrative.

- If the above criteria are not met and re-analysis is not possible, then the sample data must be qualified. This anomaly must be addressed in the project narrative.

9.3. Laboratory Control Sample (LCS) - One aqueous LCS must be processed with each preparation batch. The LCS must contain all analytes of interest and must be carried through the entire analytical procedure. Aqueous LCS spike levels are provided in Table II (Appendix A). The LCS is used to monitor the accuracy of the analytical process. Ongoing monitoring of the LCS results provides evidence that the

laboratory is performing the method within acceptable accuracy and precision guidelines.

- If any analyte is outside established control limits, the system is out of control and corrective action must occur. Unless in-house control limits are established, a control limit of 80 - 120% recovery must be applied.

- In the event that an MS/MSD analysis is not possible, a Laboratory Control Sample Duplicate (LCSD) must be analyzed. The LCSD recovery is evaluated using the same control limits as the LCS. The RPD of the LCS and LCSD must be compared to in-house limits.

- In the instance where the LCS recovery is greater than the upper control limit and the sample results are < RL, the data may be reported with qualifiers. Such action must be addressed in the report narrative.

- Corrective action will be repreparation and re-analysis of the batch unless the client agrees that other corrective action is acceptable. For Ohio VAP projects the corrective action must be repreparation and reanalysis of the batch.

9.4 Additional information on QC samples can be found in QA Policy QA-003. Ohio VAP projects must reference this SOP instead of policy QA-003 for information on QC samples.

9.5 Matrix Spike/Matrix Spike Duplicate (MS/MSD) - One MS/MSD pair must be processed for each preparation batch. A matrix spike (MS) is a field sample to which known concentrations of target analytes have been added. A matrix spike duplicate (MSD) is a second aliquot of the same sample (spiked identically as the MS) prepared and analyzed along with the sample and matrix spike. Some client-specific data quality objectives (DQOs) may require the use of sample duplicates in place of, or in addition to, MS/MSDs. The MS/MSD results are used to determine the effect of a matrix on the precision and accuracy of the analytical process. Due to the potential variability of the matrix of each sample, these results may have immediate bearing only on the specific sample spiked. Samples identified as field blanks cannot be used for MS/MSD analysis. Spiking levels are provided in Tables II and V (Appendix A).

- If any analyte recovery or RPD falls outside the acceptance range, the recovery of that analyte must be in control for the LCS. For Methods 200.7, 6010A, and 6010B, control limits of 75-125% recovery and 20% RPD or historical acceptance criteria must be applied to the MS/MSD. If the LCS recovery is within limits, then the laboratory operation is in control and the results may be accepted. If the recovery of the LCS is outside limits, corrective action must be taken. Corrective action will include repreparation and re-analysis of the batch. MS/MSD results, which fall outside the control limits, must be addressed in the narrative.

- If the native analyte concentration in the MS/MSD exceeds four times the spike level for that analyte, the recovery data is reported as NC, MSB (i.e., not

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calculated). Two other narrative notes for metals analyses: Matrix spike/spike duplicate spike recovery/recoveries was/were outside the acceptance limits of some analytes. The acceptable LCS analysis data indicated that the analytical system was operating within control and this condition is most likely due to matrix interference. See the Matrix Spike Report for the affected analytes which will be flagged with N. Matrix spike/spike duplicate relative percent difference (RPD) exceeded the acceptance limits for some analytes. The imprecision may be attributed to sample heterogeneity. See the Matrix Spike Report for the affected analytes, which will be flagged with *.

- If an MS/MSD is not possible due to limited sample volume, then a laboratory control sample duplicate (LCSD) must be analyzed. The RPD of the LCS and LCSD must be compared to the matrix spike RPD limits.

9.6 Dilution test – A dilution test is performed to determine whether significant physical or chemical interferences exist due to the sample matrix. One sample per preparation batch must be processed as a dilution test. The test is performed by running a sample at a 5 times (1:4) dilution. Samples identified as field blanks cannot be used for dilution tests. The results of the diluted sample after correction for dilution must agree within 10% of the original sample determination when the original sample concentration is greater than 50 times the IDL. If the results are not within 10%, the possibility of chemical or physical interference exists and the data is flagged.

9.12 Control Limits

9.12.1 Control limits are established by the laboratory as described in SOP NC-QA-018.

9.12.2 Laboratory control limits are internally generated and updated periodically unless method specified. Control limits are easily accessible via LIMS (QC Browser program).

9.13 Method Detection Limits (MDLs) and MDL Checks

9.13.1 MDLs and MDL Checks are established by the laboratory as described in SOPs NC-QA-021 and CA-Q-S-006.

9.13.2 MDLs are easily accessible via LIMS (QC Browser program)

9.14 Nonconformance and Corrective Action

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9.14.1 Any deviations from QC procedures must be documented as a nonconformance with applicable cause and corrective action approved by the facility QA Manager.

10. CALIBRATION AND STANDARDIZATION

10.1. Set up the instrument with the operating parameters recommended by the manufacturer. Allow the instrument to become thermally stable before beginning calibration (approximately 30 minutes of warm-up is required). Refer to the instructions in Appendix F.

10.3 Profile and calibrate the instrument according to the instrument manufacturer's recommended procedures. Flush the system with the calibration blank between each standard or as the manufacturer recommends. The calibration curve must consist of a minimum of a blank and a standard. Refer to Appendix F for detailed setup and operation protocols. Refer to Instruction Manuals in laboratory Calibration must be performed daily and each time the instrument is set up. Instrument runs may be continued over periods exceeding 24 hours as long as all calibration verification (CCV) and interference check QC criteria are met. The instrument standardization date and time must be included in the raw data

10.4 Initial Calibration Verification (ICV/ICB) - Calibration accuracy is verified by analyzing a second source standard (ICV). For analyses conducted under Method 200.7, the ICV result must fall within 5% of the true value for that solution with relative standard deviation <3% from replicate (minimum of two) exposures. For Method 6010B, the ICV must fall within 10% of the true value for that solution. For Method 6010B, the relative standard deviation must be <5% from replicate (minimum of two) exposures. An ICB is analyzed immediately following the ICV to monitor low level accuracy and system cleanliness. The ICB result must fall within ± the RL from zero. If either the ICV or ICB fail to meet criteria, the analysis must be terminated, the problem corrected, the instrument recalibrated, and the calibration reverified (see Sections 11.6 through 11.8 for required run sequence).

10.5Continuing Calibration Verification (CCV/CCB) - Calibration accuracy is monitored throughout the analytical run through the analysis of a known standard after every 10 samples and at the end of the sample run. The CCV is to be a mid-range standard made from a dilution of the calibration standard. The CCV for all methods must fall within 10% of the true value for that solution. For Methods 6010B, and 200.7, the relative standard deviation must be <5% from replicate (minimum of two) exposures. A CCB is analyzed immediately following each CCV (see Sections 11.6 through 11.8 for required run sequence). The CCB result must fall within ± RL from zero. Sample results may only be reported when bracketed by valid CCV/CCB pairs. If a mid-run CCV or CCB fails, all affected samples will be re-analyzed with valid CCV/CCB pairs (refer to Section 11.7 for an illustration of the appropriate rerun sequence). Exceptions: If CCB > RL, samples < RL can be reported with an NCM. If CCV is outside of criteria on the high side, samples < RL can be reported with an NCM.

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10.6 Verification Standards (6010A only). The calibration standards are analyzed against the calibration curve and must be within 5% of the true value.

10.7 Interference Check Analysis (ICSA/ICSAB) - The validity of the inter-element correction factors is demonstrated through the successful analysis of interference check solutions. The ICSA contains only interfering elements, the ICSAB contains analytes and interferents. Refer to Table IV (Appendix A) for the details of ICSA and ICSAB composition. Custom multi-element ICS solutions must be used. All analytes must be spiked into the ICSAB solution; therefore, if a non-routine analyte is required, then it must be manually spiked into the ICSAB using a certified ultra high purity single element solution or custom lab-specific mix. If the ICP will display overcorrection as a negative number, then the non-routine elements can be controlled from the ICSA as described in Section 10.6.3. Elements known to be interferents on a required analyte must be included in the ICP run when that analyte is determined. Aluminum, iron, calcium, and magnesium must always be included in all ICP runs.

10.7.1 The ICSA and ICSAB solutions must be run at the beginning of the run (see Sections 11.6 or 11.7 for required run sequence). For Method 6010A, the ICSA and ICSAB solutions must be run every 8 hours.

10.7.2 The ICSAB results for the interferents must fall within 80 - 120% of the true value. If any ICSAB interferent result fails criteria, the analysis must be terminated, the problem corrected, the instrument recalibrated, and the samples rerun.

10.7.3 ICSA results for the non-interfering elements with reporting limits ≤ 10 ug/L must fall within ± 2 times the RL from zero. ICSA results for the non-interfering elements with RLs > 10 µg/L must fall within ± 1 times the RL from zero. If the ICSA results for the non-interfering elements do not fall within ± two times RL (RL ≤10) or ± 1xRL (RL>10) from zero, the field sample data must be evaluated as follows.

- If the non-interfering element concentration in the ICSA is the result of contamination versus a spectral interference, and this reason is documented, the field sample data can be accepted.
- If the affected element was not required, then the sample data can be accepted.
- If the interfering elements are not present in the field sample at a concentration which would result in a false positive or negative result greater than ± two times the RL from zero, then the field sample data can be accepted.
- If the interfering element is present in the field sample at a level which would result in a false analyte signal greater than ± two times the RL from

zero, the data can be accepted only if the concentration of the affected analyte in the field sample is more than ten times the analyte signal in the ICSA.

- If the data does not meet the above conditions, then the IECs must be re-evaluated and corrected if necessary and the affected samples re-analyzed or the sample results manually corrected through application of the new IEC to the raw results. If the results are recalculated manually, the calculations must be clearly documented on the raw data.

10.8 CRI - To verify linearity near the RL for ICP analysis, a CRI standard is run at the beginning of each sample analysis run. Additionally, some projects may require CRI analysis at the end of the run (see Sections 11.6 or 11.7 for required run sequence). Evaluate associated samples based upon advisory limits of ± 50% of true value. This standard is required for Method 6010A.

Note: The custom CRI mix contains most analytes at a level near the standard lab reporting limit.

11. PROCEDURE

11.1. A minimum of two exposures for each standard, field sample and QC sample is required. The average of the exposures is reported. For Trace ICP analyses, the results of the sum channel must be used for reporting.

11.2. Prior to calibration and between each sample/standard, the system is rinsed with the calibration blank solution.

11.3. The use of automated QC checks through the instrument software is highly recommended for all calibration verification samples (ICV,CCV), blanks (ICB,CCB,PB), interference checks (ICSA,ICSAB), and field samples (linear range) to improve the data review process.

11.4. To facilitate the early identification of QC failures and samples requiring rerun, it is strongly recommended that sample data be reviewed periodically throughout the run.

11.5. The following procedural guidelines must be followed when using an internal standard:

11.5.1 Typically used internal standard is: yttrium. (Note: Any element can be used that is not typically found in environmental samples at a high rate of occurrence.)

11.5.2 The internal standard (IS) must be added to every sample and standard at the same concentration. It is recommended that the IS be added to each analytical sample automatically through use of a third pump channel and mixing coil. Internal standards must be added to blanks, samples, and

standards in a like manner, so that dilution effects resulting from the addition may be disregarded.

11.5.3 The concentration of the internal standard must be sufficiently high to obtain good precision in the measurement of the IS analyte used for data correction and to minimize the possibility of correction errors if the IS analyte is naturally present in the sample.

11.5.4 The internal standard raw intensity counts must be printed on the raw data.

11.5.5 The analyst must monitor the response of the internal standard throughout the sample analysis run. This information is used to detect potential problems and identify possible background contributions from the sample (i.e., natural occurrence of IS analyte). The instrument automatically adjusts sample results based on comparison of the internal standard intensity in the sample to the internal standard intensity at calibration.

11.5.5.1 If the internal standard counts fall within ±30% of the counts observed in the ICB, then the data is acceptable.

11.5.5.2 If the internal standard counts in the field samples are more than ± 30% higher than the expected level, evaluate the run to determine if a dilution is needed due to matrix interference.

11.6 The following analytical sequence must be used for Methods 6010B, and 200.7:

Instrument Calibration
ICV
ICB
CRI
ICSA
ICSAB
CCV
CCB
10 samples
CCV
CCB
10 samples
CCV
CCB
Repeat sequence of up to 10 samples between CCV/CCB pairs as required to complete run
CRI (The CRI counts as a sample analysis.)
CCV
CCB

Refer to Quality Control Section 9.0 and Table VI (Appendix A) for Methods 6010A,

6010B, and 200.7 quality control criteria.

11.7 The following analytical sequence must be used for Methods 6010A:

Instrument Calibration
ICV
ICB
CRI (+/- 50%)
Calibration verification standards
ICSA (maximum every 8 hours)
ICSAB (maximum every 8 hours)
CCV
CCB
10 samples
CCV
CCB
10 samples
CCV
CCB
Repeat sequence of up to 10 samples between CCV/CCB pairs as required to complete run
CRI (The CRI counts as a sample analysis.)
CCV
CCB

11.8 The following run sequence provides an illustration of a mid-run CCV or CCB failure and the appropriate corrective action run sequence as described in Section 10.4.

Original Run: Instrument Calibration
ICV
ICB
CRI
ICSA
ICSAB
CCV
CCB
10 samples
CCV
CCB
10 samples
CCV
CCB
10 samples **
CCV * * Failure occurs at CCV/CCB
CCB * **Samples requiring rerun for affected analytes
10 samples **

CCV
CCB
10 samples
CCV
CCB

11.9 The instrument may be reprofiled between CCV/CCB pairs to correct for environment- induced drift.

11.10 Guidelines are provided in the Appendix C, D, and E on procedures to minimize contamination of samples and standards, preventive maintenance, and troubleshooting.

11.11 All measurements must fall within the defined linear range where spectral interference correction factors are valid. Dilute and reanalyze all samples for required analytes that exceed the linear range. If an inter-element correction exists for an analyte, which exceeds the linear range, the IEC may be inaccurately applied. Therefore, even if an overrange analyte may not be required to be reported for a sample, if that analyte is an interferent for any requested analyte in that sample, the sample must be diluted. Acid strength must be maintained in the dilution of samples.

11.12 Any variation in procedure must be completely documented using instrument run logs, maintenance logs, report narratives, a Nonconformance Memo, or an anomaly report; and is approved by a Supervisor/Group Leader and QA Manager.

11.13 Nonconformance documentation must be filed in the project file.

11.14 Any unauthorized deviations from this procedure must also be documented as a nonconformance with a cause and corrective action described.

11.15 Analytical Documentation

11.15.1 Record all analytical information in the analytical logbook/logsheet which may be in an electronic format, including the analytical data from standards, blanks, LCSs, MS/MSDs, and any corrective actions or modifications to the method.

11.15.2 All standards are logged into a department standard logbook. All standards are assigned a unique number for identification. Logbooks are reviewed by the supervisor or designee.

11.15.3 Documentation, such as all associated instrument printouts (final runs, screens, reruns, QC samples, etc.) and daily calibration data corresponding to all final runs, is available for each data file.

11.15.4 Sample results and associated QC are entered into the LIMs after final

technical review.

12 DATA ANALYSIS AND CALCULATIONS

12.1. ICV percent recoveries are calculated according to the equation:

$$\%R = 100\left(\frac{Found(ICV)}{True(ICV)}\right)$$

12.2. CCV percent recoveries are calculated according to the equation:

$$\%R = 100\left(\frac{Found(CCV)}{True(CCV)}\right)$$

12.3. Matrix Spike Recoveries are calculated according to the following equation:

$$\%R = 100\left(\frac{SSR - SR}{SA}\right)$$

Where:
SSR = Spike Sample Result
SR = Sample Result
SA = Spike Added

12.4. The relative percent difference (RPD) of matrix spike/matrix spike duplicates are calculated according to the following equations:

$$RPD = 100\left[\frac{|MSD - MS|}{\left(\frac{MSD + MS}{2}\right)}\right]$$

Where:
MS = determined spiked sample concentration
MSD = determined matrix spike duplicate concentration

12.5. The final concentration for a digested aqueous sample is calculated as follows:

$$mg/L = \frac{C \times V1 \times D}{V2}$$

Where:

C = Concentration (mg/L) from instrument readout (mean of two exposures)
D = Instrument dilution factor
V1 = Final volume in liters after sample preparation
V2 = Initial volume of sample digested in liters

12.6. The final concentration determined in digested solid samples is calculated as follows:

$$mg / Kg, dry\ weight = \frac{C \times V \times D}{W}$$

Where:
C = Concentration (mg/L) from instrument readout (mean of two exposures)
D = Instrument dilution factor
V = Final volume in liters after sample preparation
W = Weight in Kg of wet sample digested

12.7. The LCS percent recovery is calculated according to the following equation:

$$\%R = 100\left(\frac{Found(LCS)}{True(LCS)}\right)$$

12.8. The dilution test percent difference for each component is calculated as follows:

$$\%Difference = \frac{|I - S|}{I} \times 100$$

Where:
I = Sample result (Instrument reading)
S = Dilution test result (Instrument reading × 5)

12.9. Appropriate factors must be applied to sample values if dilutions are performed.

12.10. Trivalent Chromium

12.10.1. Trivalent chromium (CR^{+3}) is the result obtained by subtracting the hexavalent chromium (CR^{+6}) results for a sample from the total chromium result from that sample. The total chromium result is determined using the procedures in this SOP. The hexavalent chromium result is determined using the procedures in TestAmerica North Canton SOP NC-WC-024.

12.10.2. Reporting Limits

12.10.2.1. The TestAmerica North Canton water reporting limit for trivalent chromium is 0.02 mg/l.

12.10.2.2. The TestAmerica North Canton solid reporting limit for trivalent chromium is 2.0 mg/kg, wet weight.

12.10.3. Calculations: Cr^{+3} = Cr, total – Cr^{+6}

13. METHOD PERFORMANCE

13.1. Each laboratory must have initial demonstration of performance data on file for each analyte of interest as described in Section 9.0.

13.2. Refer to Tables I and IA in Appendix A for the list of analytes that may be analyzed using this SOP.

13.3. Method performance is determined by the analysis of MS and MSD samples as well as method blanks and laboratory control samples. The MS or MSD recovery must fall within ± 25 % and the MS/MSD must compare within 20% RPD or within the laboratory's historical acceptance limits. These criteria apply to analyte concentrations greater than or equal to ten times the IDL. Method blanks must meet the criteria specified in Section 9.2. The laboratory control samples must recover within 20% of the true value or within the laboratory's historical acceptance limits.

13.4. Training Qualification

13.4.1 The Group/Team Leader or the Supervisor has the responsibility to ensure that this procedure is performed by an associate who has been properly trained in its use and has the required experience.

14. POLLUTION PREVENTION

14.2. It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the corporate environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention".

15. WASTE MANAGEMENT

15.1. All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention."

15.2. Waste Streams Produced by this Method

15.2.1. The following waste streams are produced when this method is carried out:

15.2.2. Acid waste consisting of sample and rinse solution - Any sample waste generated must be collected and disposed of in the acid waste drum located in the Metals Lab.

15.2.3. Standards must be purchased and prepared in volumes consistent with laboratory use to minimize the volume of expired standards to be disposed.

16. REFERENCES

16.1. References

16.1.1. 40 CFR Part 136, Appendix B, 7-5-95, Determination of Method Detection Limits

16.1.2. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition, Final Update I, July 1992. Method 6010A.

16.1.3. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition, Final Update III, Revision 2, December 1996. Method 6010B.

16.1.4. Determination of Metals and Trace Elements in Water and Wastes by Inductively Coupled Plasma-Atomic Emission Spectrometry, Revision 4.4, May 1994. Method 200.7

16.1.5. Inductively Coupled Plasma – Atomic Emission Spectrometric Method for Trace Element Analysis of water and wastes Method 200.7, 40 CFR – Chapter I – Part 136 – Appendix C. Electronic version dated September 30, 2002

16.1.6. TestAmerica North Canton Quality Assurance Manual (QAM), current version

16.1.7. TestAmerica Corporate Environmental Health and Safety Manual, CW-E-M-001, and TestAmerica North Canton Facility Addendum and Contingency Plan, current version

16.1.8. Revision History

Historical File:		Revision 2.0: 10/27/97		Revision 0: 01/08/04 (NC-MT-012)
(formerly CORP-MT-0001NC)		Revision 2.1: 04/19/99		
		Revision 3.1: 10/04/00		
		Revision 3.2: 01/19/01		
		Revision 3.3: 12/05/01		

		Revision 3.4: 01/08/04		

16.2. Associated SOPs and Policies, latest version

16.2.1. TestAmerica North Canton QC Program, QA-003

16.2.2. Statistical Evaluation of Data and Development of Control Charts, NC-QA-018

16.2.3. Method Detection Limits and Instrument Detection Limits, NC-QA-021 and CA-Q-S-006

16.2.4. Supplemental Practices for DoD Project Work, NC-QA-016

16.2.5. Hexavalent Chromium (Colorimetric), NC-WC-024

16.2.6. Acid Digestion of Soils, SW846 Method 3050B, NC-IP-010

16.2.7. Acid Digestion of Aqueous Samples by SW846 and MCAWW 200 Series Methods, NC-IP-011Standards and Reagents, NC-QA-017

17. MISCELLANEOUS (TABLES, APPENDICES, ETC.)

17.1. Modifications/Interpretations from reference method

17.1.1. Modifications/interpretations from Methods 6010B and 200.7.

17.1.1.1. TestAmerica North Canton Laboratories use mixed calibration standard solutions purchased from approved vendors instead of using individual mixes prepared in-house as recommended by the subject methods.

17.1.1.2. Methods 200.7 and 6010B state that if the correction routine is operating properly, the determined apparent analyte(s) concentration from analysis of each interference solution must fall within a specific concentration range around the calibration blank. In determining IECs because of lack of definition clarification for "concentration range around the calibration blank," TestAmerica North Canton has adopted the procedure in EPA CLP ILMO4.0.

17.1.1.3. Whenever a new or unusual matrix is encountered, a series of tests be performed prior to reporting concentration data for that analyte. The dilution test helps determine if a chemical or physical

interference exists. Because TestAmerica North Canton laboratories receive no prior information from clients regarding when to expect a new or unusual matrix, TestAmerica North Canton may select to perform a dilution test on one sample in each prep batch. According to the method, the post digestion spike (PDS) determines any potential matrix interferences. At TestAmerica North Canton, matrix interference is determined by evaluating data for the LCS and MS/MSD. TestAmerica North Canton REQUIRES documented, clear guidance when a new or unusual matrix will be received for a project and a request to perform the dilution test or PDS on a client-identified sample.

17.1.2. Modifications from Method 200.7

17.1.2.1. Method 200.7 defines the IDL as the concentration equivalent to a signal due to the analyte, which is equal to three times the standard deviation of a series of ten replicate measurements of the calibration blank signal at the same wavelength. TestAmerica North Canton labs utilize the IDL definition as defined in Section 9.1 of this SOP.

17.1.2.2. The calibration blank is prepared in an acid matrix of 5% HNO_3/5% HCl instead of the specified 2% HNO_3/10% HCl matrix as the former matrix provides for improved performance relative to the wide variety of digestate acid matrices which result from the various EPA preparation protocols applied.

17.1.2.3. Method Section 9.3.4 specifies that "Analysis of the ICV (ICSA/AB) solution immediately following calibration must verify that the instrument is within ± 5% of calibration with a relative standard deviation <3% from replicate integrations ≥ 4". TestAmerica North Canton uses a minimum of two exposures.

17.1.2.4. The 40 CFR version of Method 200.7 requires the instrument check standard to agree within ± 5% of expected values. Also, the 40 CFR version requires the interference check sample to be analyzed at the beginning, end, and at periodic intervals throughout the sample run. At TestAmerica North Canton, the instrument check standard equals the CCV, which must agree within ± 10% of expected values, and the ICSA standards are analyzed only at the beginning of a sample run. TestAmerica's procedures are in line with the Rev. 4.4, May 1994 version of Method 200.7.

17.1.2.5. Section 7.12 of Method 200.7 indicates that the QCS (ICV) must be prepared at a concentration near 1 ppm. The ICV specified in this SOP accommodates the 1 ppm criteria for the majority of analytes. For the remaining analytes, this SOP specifies ICV concentrations

which are appropriate to the range of calibration. The intent of the ICV, verification of calibration standard accuracy, is independent of the ICV concentration used.

17.1.2.6. The ICS criteria applied by this SOP differ from those stated in the method. Method 200.7 Section 10.4 states that results must fall within the established control limits of 3 times the standard deviation of the calibration blank for that analyte. The control limits listed in this SOP are those applicable to the EPA designed solution.

17.1.2.7. Method 200.7 Section 9.3.4 states the CCB must be less than the IDL, but > the lower 3-sigma control limit of the calibration blank. The intent of this requirement is to ensure that the calibration is not drifting at the low end. TestAmerica North Canton has adopted an absolute control limit of ± RL from zero for calibration blank criteria. SOP Section 10.4 provides the detailed corrective action criteria that must be followed.

17.1.3. Modifications from Method 6010B

17.1.3.1. Chapter 1 of SW-846 states that the method blank must not contain any analyte of interest at or above the MDL. This SOP states that the method blank must not contain any analyte of interest at or above the reporting limit. Common lab contaminants are allowed up to two times the reporting limit in the blank following consultation with the client.

17.1.3.2. Method 6010B Section 8.6.1.3 states that the results of the calibration blank are to agree within three times the IDL. If not, repeat the analysis two or more times, and average the results. If the average is not within three standard deviations of the background mean, terminate the analysis, correct the problem, recalibrate, and re-analyze the previous ten samples. The intent of this requirement is to ensure that the calibration is not drifting at the low end. TestAmerica North Canton has adopted an absolute control limit of ± RL from zero for calibration blank criteria. See SOP Section 10.4 for a detailed description of the required corrective action procedures.

17.1.4 Modifications from Method 6010A

17.1.4.1 Chapter 1 of SW846 states the method blank should not contain any analyte of interest at, or above, the MDL. This SOP states the method blank must not contain any analyte of interest at, or above, the reporting limit. Common lab contaminants are allowed up to two times the reporting limit in the blank.

17.1.4.2 Calibration is performed according to instrument manufacturer's recommendations. Method 6010A provides contradictory instructions for instrument calibration by stating to calibrate both according to instrument manufacturer's recommendations as well as to use a blank and three standards. TJA has stated that the use of multiple standards may be detrimental to determinations near the detection limit due to the inability of the linear equation to force-fit through the origin, and recommends the calibration be performed using a blank and one standard. This SOP requires verification of the initial instrument calibration using a CRI at two times the RL, an ICV at 5 - 25% of calibration level, a CCV at 50% of calibration level, and by rerunning the high calibration standard (HCAL) post calibration to demonstrate linearity (See Tables I and IA).

17.1.4.3 Section 5.6 of Method 6010A states the instrument check standard (CCV) should be prepared from a second source standard. This SOP states the CCV will be from the same source as the calibration standards. The purpose of the second source standard is to verify the accuracy of the calibration standards. The intent of this requirement is met through the analysis of a second source ICV standard prior to the analysis of samples. The use of a same source CCV provides for a more accurate and consistent measure of instrument drift from initial calibration.

17.1.4.4 Section 5.7 of Method 6010A states spiking of the ICS solution with analytes is not required if the ICP will display over-correction as a negative number. All TestAmerica instrumentation has this capability, and therefore, the spike analysis is not required. TestAmerica does run a spiked ICSAB, but the analytes are not spiked at the 10x IDL level referenced in Method 6010A. The ICSAB solution run by TestAmerica is based on the design of the ICSA/ICSAB solution provided by EPA directly to contract environmental labs.

17.1.4.5 Method 6010A uses a Quality Control Standard (QCS) on a weekly basis to verify calibration standard accuracy. TestAmerica refers to QCS as an ICV, and the accuracy verification is performed on a daily basis. The QCS described in Method 6010A is made to contain analytes at 10x the IDL. The TestAmerica ICV solution is not made at 10x IDL for all elements as this concentration is not appropriate relative to the standard reporting limits. TestAmerica designed the ICV to be a reliable indicator of calibration standard accuracy by raising the analyte concentrations to a level where the analytical determination is not impacted by low-level curve bias.

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17.1.4.6 Method 6010A states the CCB should be within three times the standard deviations of the average blank reading. The intent of this requirement is to ensure the calibration is not drifting at the low end. In the absence of guidance on how to determine the mean blank level from EPA, TestAmerica has adopted an absolute control limit of ± RL from zero for calibration blank criteria.

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APPENDIX A

TABLES

TABLE I. Methods 200.7, 6010A, and 6010B Target Analyte List

Element	Symbol	CAS #	Reporting Limit (ug/L) Water	Reporting Limit (mg/kg) Soil
Aluminum	Al	7429-90-5	200	20
Antimony	Sb	7440-36-0	60	6
Arsenic	As	7440-38-2	300	30
Barium	Ba	7440-39-3	200	20
Beryllium	Be	7440-41-7	5.0	0.5
Boron	B	7440-42-8	200	20
Cadmium	Cd	7440-43-9	5.0	0.5
Calcium	Ca	7440-70-2	5000	500
Chromium	Cr	7440-47-3	10	1
Cobalt	Co	7440-48-4	50	5
Copper	Cu	7440-50-8	25	2.5
Iron	Fe	7439-89-6	100	10
Lead	Pb	7439-92-1	100	10
Magnesium	Mg	7439-95-4	5000	500
Manganese	Mn	7439-96-5	15	1.5
Molybdenum	Mo	7439-98-7	40	4
Nickel	Ni	7440-02-0	40	4
Potassium	K	7440-09-7	5000	500
Selenium	Se	7782-49-2	250	25
Silver	Ag	7440-22-4	10	1
Sodium	Na	7440-23-5	5000	500
Thallium	Tl	7440-28-0	2000	200
Vanadium	V	7440-62-2	50	5
Zinc	Zn	7440-66-6	20	2
Tin	Sn	7440-31-5	100	10
Titanium	Ti	7440-32-6	50	5

TABLE IA. Method 200.7, 6010A, and 6010B Trace ICP Target Analyte List

Element	Symbol	CAS #	Reporting Limit (ug/L) Water	Reporting Limit (mg/kg) Soil
Arsenic	As	7440-38-2	10	1.0
Lead	Pb	7439-92-1	3.0	0.3
Selenium	Se	7782-49-2	5.0	0.5
Thallium	Tl	7440-28-0	10	1.0
Antimony	Sb	7440-36-0	10	1.0
Cadmium	Cd	7440-43-9	2.0	0.2
Silver	Ag	7440-22-4	5.0	0.5
Chromium	Cr	7440-47-3	5.0	0.5

TABLE II. Matrix Spike and Aqueous Laboratory Control Sample Levels

ELEMENT	Concentration (ug/L)
Aluminum	2000
Antimony	500
Arsenic	2000
Barium	2000
Beryllium	50
Cadmium	50
Calcium	50000
Chromium	200
Cobalt	500
Copper	250
Iron	1000
Lead	500
Magnesium	50000
Manganese	500
Molybdenum	1000
Nickel	500
Potassium	50000
Selenium	2000
Silver	50
Sodium	50000
Thallium	2000
Vanadium	500
Zinc	500
Boron	1000
Tin	2000
Titanium	1000

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TABLE III . Trace ICP Calibration and Calibration Verification Standards

Element	Calibration Level	RL (ug/L)	ICV (ug/L)	CCV (ug/L)
Aluminum	50000	200	12500	25000
Antimony	1000	10	250	500
Arsenic	1000	10	250	500
Barium	4000	10	1000	2000
Beryllium	4000	5	1000	2000
Cadmium	1000	2	250	500
Calcium	100000	5000	25000	50000
Chromium	4000	5	1000	2000
Cobalt	4000	50	1000	2000
Copper	4000	25	1000	2000
Iron	50000	100	12500	25000
Lead	1000	3	250	500
Magnesium	100000	5000	25000	50000
Manganese	4000	15	1000	2000
Molybdenum	4000	40	1000	2000
Nickel	4000	40	1000	2000
Potassium	100000	5000	25000	50000
Selenium	1000	5	250	500
Silver	2000	5	500	1000
Sodium	100000	5000	25000	50000
Thallium	2000	10	500	1000
Vanadium	4000	50	1000	2000
Zinc	4000	20	1000	2000
Boron	10000	200	1000	5000
Tin	10000	100	1000	5000
Titanium	10000	50	1000	5000

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TABLE IV. Interference Check Sample Concentrations

Element	ICSA (ug/L)	ICSAB (ug/L)
Aluminum	500000	500000
Antimony	-	1000
Arsenic	-	1000
Barium	-	500
Beryllium	-	500
Cadmium	-	1000
Calcium	500000	500000
Chromium	-	500
Cobalt	-	500
Copper	-	500
Iron	200000	200000
Lead	-	1000
Magnesium	500000	500000
Manganese	-	500
Molybdenum	-	1000
Nickel	-	1000
Potassium	-	10000
Selenium	-	1000
Silver	-	1000
Sodium	-	10000
Thallium	-	1000
Vanadium	-	500
Zinc	-	1000
Tin	-	1000
Boron		1000
Titanium		1000

TABLE V. TCLP Reporting Limits, Regulatory Limits and Matrix Spike Levels

ELEMENT	Reporting Level (ug/L)	Regulatory Limit (ug/L)	Spike Level (ug/L)
Arsenic	500	5000	5000
Barium	10000	100000	50000
Cadmium	100	1000	1000
Chromium	500	5000	5000
Lead	500	5000	5000
Selenium	250	1000	1000
Silver	500	5000	1000

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TABLE VI. Summary of Quality Control Requirements

QC Parameter	Frequency *	Acceptance Criteria	Corrective Action
Two-point Initial Calibration	Beginning of every analytical run, every 24 hours, whenever instrument is modified, or CCV criterion is not met		Terminate analysis. Correct the problem. Prepare new standards. Recalibrate following system performance.
ICV	Beginning of every analytical run.	Method 200.7: 95 - 105 % recovery RSD dupl. exp < 3% Method 6010A: 90 - 110 % recovery Method 6010B: 90 - 110 % recovery RSD dupl. exp < 5%	Terminate analysis. Correct the problem. Recalibrate.
ICB	Beginning of every analytical run, immediately following the ICV.	The result must be within ± RL from zero.	Terminate analysis. Correct the problem. Recalibrate.
CCV	Every 10 samples and at the end of the run.	Method 200.7 & 6010B:90 - 110 % recovery RSD dupl. exp < 5% Method 6010A: 90 - 110 % recovery	Terminate analysis. Correct the problem. Recalibrate and rerun all samples not bracketed by acceptable CCV. Samples < RL can be accepted if the CCV is outside on high side with NCM.
CCB	Immediately following each CCV	The result must be within ± RL from zero	Terminate analysis. Correct the problem. Recalibrate and rerun all samples not bracketed by acceptable CCB. Samples < RL can be accepted if the CCB is outside on high side with NCM.
CRI	Beginning of every run	50-150% recovery (advisory) (required for 6010A)	Evaluate associated samples.
Calibration verification	After the CRI	+/- 5%	Recalibrate

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QC Parameter	Frequency *	Acceptance Criteria	Corrective Action
(6010A only)			
ICSA	Beginning of every run (maximum every 8 hours for 6010A)	See Section 9.9.3	See Section 9.9.3
ICSAB	Immediately following each ICSA (maximum every 8 hours for 6010A)	Results must be within 80 - 120% recovery	See Section 9.9.2.
Dilution Test	One per prep batch	For samples > 50 times the IDL, dilutions must agree within 10%	Narrate the possibility of physical or chemical interference per client request.
Method Blank	One per sample preparation batch of up to 20 samples	The result must be less than or equal to the RL. Common lab contaminants may be accepted up to 2x the RL (see Section 9.2). (not applicable for Ohio VAP) Sample results greater than 20 times the blank concentration are acceptable. (not applicable for Ohio VAP) Samples for which the contaminant is < RL may not require redigestion or re-analysis (see Section 9.2).	Redigest and re-analyze samples. Note exceptions under criteria section. See Section 9.2 for additional requirements.
Laboratory Control Sample (LCS)	One per sample preparation batch of up to 20 samples	Aqueous LCS must be within 80 - 120% recovery or in-house control limits. Samples for which the contaminant is < RL and the LCS results are > 120% may not	Terminate analysis. Correct the problem. Re-digest and re-analyze all samples associated with the LCS.

QC Parameter	Frequency *	Acceptance Criteria	Corrective Action
		require redigestion or re-analysis (see Section 9.3)	
Matrix Spike	One per sample preparation batch of up to 20 samples	75 - 125 % recovery or in-house control limits	In the absence of client specific requirements, flag the data. No flag required if sample level is > 4 times the spike added.
Matrix Spike Duplicate	See Matrix Spike	75 - 125 % recovery; RPD ≤ 20%	See Corrective Action for Matrix Spike.

* See Sections 11.6, 11.7, and 11.8 for exact run sequence to be followed.

APPENDIX B CROSS REFERENCE OF TERMS COMMONLY USED IN METHODS EPA 200.7, SW 6010A/6010B, AND TESTAMERICA NORTH CANTON SOP

EPA 200.7	SW 6010A/6010B	TestAmerica North Canton SOP
Calibration blank (CB)	Calibration blank	Initial and continuing calibration blanks (ICB/CCB)
Dilution test	Dilution test	Dilution Test
Instrument detection limit (IDL)	Instrument detection limit (IDL)	Instrument detection limit (IDL)
Instrument performance check (IPC)	Continuing calibration verification (CCV)	Continuing calibration verification (CCV)
Internal standard	Internal standard	Internal standard (IS)
Laboratory duplicates	N/A	N/A
Laboratory fortified blank (LFB)	N/A	Laboratory control sample (LCS)
Laboratory fortified sample matrix (LFM)	Matrix spike and matrix spike duplicate (MS/MSD)	Matrix spike and matrix spike duplicate (MS/MSD)
Laboratory reagent blank (LRB)	Method blank	Method or Prep blank (MB)
Linear dynamic range (LDR)	Linear dynamic range (LDR)	Linear dynamic range (LDR)
Method detection limit (MDL)	Method detection limit (MDL)	Method detection limit (MDL)
Quality control sample (QCS)	Check standard or Initial calibration verification (ICV)	Initial calibration verification (ICV)
Spectral interference check solution (SIC)	Interference check solution (ICS)	Interference check solution (ICSA/ICSAB)

APPENDIX C - TROUBLESHOOTING GUIDE

Problem	Possible Cause/ Solution
High Blanks	Increase rinse time Clean or replace tip Clean or replace torch Clean or replace sample tubing Clean or replace nebulizer Clean or replace mixing chamber
Instrument Drift	Replace torch (Crack) Clean or replace nebulizer (blockage) Replace pump tubing Room humidity too high Clean torch tip (salt buildup) Check for argon leaks Reprofile
Erratic Readings, Flickering Torch or High RSD	Check for argon leaks Adjust sample carrier gas Replace tubing (clogged) Check drainage (back pressure changing) Increase uptake time (too short) Increase flush time (too short) Clean nebulizer, torch or spray chamber Increase sample volume introduced Check that autosampler tubes are full Sample or dilution of sample not mixed Increase integration time (too short) Realign torch Reduce amount of tubing connectors
Standards reading twice normal absorbance or concentration	Incorrect standard used Incorrect dilution performed

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APPENDIX D - CONTAMINATION CONTROL GUIDELINES

The following procedures are strongly recommended to prevent contamination:

All glassware must be washed with detergent and tap water and rinsed with 1:1 nitric acid followed by deionized water.

Proper laboratory housekeeping is essential in the reduction of contamination in the Metals Lab. All work areas must be kept scrupulously clean.

Powdered Gloves must not be used in the Metals Lab since the powder contains silica and zinc as well as other metallic analytes. Glassware must be periodically checked for cracks and etches and discarded if found. Etched glassware can cause cross contamination of any metallic analytes.

The following are helpful hints in the identification of the source of contaminants:

Yellow pipette tips and volumetric caps can sometimes contain cadmium.

Some sample cups have been found to contain lead.

New glassware especially beakers can be a source of silica and boron.

Reagents or standards can contain contaminants or be contaminated with the improper use of a pipette.

Improper cleaning of glassware can cause contamination.

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APPENDIX E - PREVENTATIVE MAINTENANCE

A maintenance log is used to record when maintenance is performed on instruments. When an instrument problem occurs, indicate the date, time and instrument number. Then identify the problem and corrective action in the Maintenance Log.

The following procedures are required to ensure that that the instrument is fully operational:

	Change sample pump tubing and pump windings
As Needed	Check rinse solution and fill if needed
	Check waste containers and empty if needed
	Check sample capillary tubing is clean and in good condition
	Check droplet size to verify nebulizer is not clogged.
	Check sample flow for cross flow nebulizer
	Check pressure for vacuum systems
	Clean plasma torch assembly to remove accumulated deposits
	Clean nebulizer and drain chamber; keep free-flowing to maintain optimum performance
	Replace peristaltic pump tubing, sample capillary tubing and autosampler sipper probe
	Apply silicon spray on autosampler tracks
	Check water level in cool flow
	Change oil for vacuum systems
	Replace coolant water filter (may require more or less frequently depending on quality of cooling water)

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APPENDIX F ICP Operating Instructions

ICP Analysis (TJA 61E) Example

1. SETUP

a. Plasma Control Panel (enter)

b. (F1)-Startup

c. (F9)-Continue

d. (F2)-Levels

1. Change auxiliary gas to low – use space bar to toggle
2. Change nebulizer gas flow to 0.5 L/min.
3. Change pump rate to 130
4. Esc
5. Allow instrument to warm up approximately 30 minutes.

2. DEVELOPMENT

a. Methods (enter)

b. Enter method name

c. (F3)-Method Info.

d. Change file name

e. (F9)-Done

f. (F9)-Done/Keep

3. OPERATION

a. Analysis (enter)

b. (F5)-Profile

1. (F3)-Automatic
2. (F1)-Run

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3. If peak position is greater than ± 0.05 units from the center peak position, you must adjust the profile. If it is within ± 0.05 units, press (F9)-done.

4. To adjust select (F1)-CalcSS and enter current vernier position. (enter)

5. Adjust to new vernier position (F9)-done

6. Rerun profile until peak position is ± 0.05 units.

7. (F9) - Done

c. Autosampler (F9)

1. Enter method name (enter)

2. Enter autosampler table name (enter)

3. (F1) - Run

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Title: INDUCTIVELY COUPLED PLASMA – MASS SPECTROMETRY

[Method: EPA Method 200.8 and SW846 Method 6020]

Approvals (Signature/Date):

Technology Specialist	Date	Health & Safety Coordinator	Date
Quality Assurance Manager	Date	Laboratory Director	Date

This SOP was previously identified as SOP No. NC-MT-002, Rev 4.4, dated 07/30/08

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1. SCOPE AND APPLICATION

1.1. This procedure describes multi-elemental analysis by inductively coupled plasma-mass spectrometry (ICP-MS) based on SW-846 protocol as described in EPA Methods 6020 and 200.8. The source method lists 15 elements approved for analysis by ICP/MS (Al, Sb, As, Ba, Be, Cd, Cr, Co, Cu, Pb, Mn, Ni, Ag, Tl, and Zn). Additional elements may be included provided that the method performance criteria presented in Section 9 is met. However, project approval may be required from the controlling agencies for compliance testing beyond the 15 elements included in the promulgated method. Reporting limits are listed in Table VII.

1.2. The procedure is applicable to the analysis of waters (groundwaters and surface waters), soils, and wastes. Preliminary acid digestion is required for groundwater, aqueous samples, sludges, sediments, and other solid wastes for which total (acid-leachable) elements are requested. See SOPs NC-IP-010 and NC-IP-011 for preparation details.

1.3. The associated LIMS method codes are MH (6020) and QV (200.8)**.**

1.4. This document accurately reflects current laboratory Standard Operating Procedures (SOP) as of the date above. All facility SOPs are maintained and updated as necessary.

2. SUMMARY OF METHOD

2.1. Aqueous samples, digestates, or leachates are nebulized into a spray chamber where a stream of argon carries the sample aerosol through the quartz torch and injects it into an R.F. plasma. There the sample is decomposed and desolvated. The ions produced are entrained in the plasma gas, and by means of a water-cooled differentially-pumped interface, introduced into a high-vacuum chamber that houses a quadrapole mass spectrometer capable of providing a resolution better than, or equal to, 0.9 AMU peak width at 10% of the peak height. For analysis by Method 200.8, the resolution requirement is 1.0 amu at 5% peak height. The ions are sorted according to their mass-to-charge ratio and measured with a channel electron multiplier. Interference must be assessed and valid corrections applied, or the data flagged, to indicate problems. Interference correction must include compensation for background ions contributed by the plasma gas, reagents and the constituents of the sample matrix. Use of the internal standard technique is required to compensate for suppressions and enhancements caused by sample matrices.

3. DEFINITIONS

3.1. Refer to the glossary in the TestAmerica North Canton Quality Assurance Manual (QAM), current version.

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4. INTERFERENCES

4.1. Isobaric Interferences. Isobaric interferences in the ICPMS are caused by isotopes of different elements forming ions with the same nominal mass-to-charge ratio (m/z). Most interferences of this type are corrected for by the instrument software.

4.2. Isobaric Molecular and Doubly Charged Ion Interferences. Isobaric molecular interferences are caused by ions consisting of more than one atom or charge. When these interferences cannot be avoided by the use of another isotope with sufficient natural abundance, corrections must be applied; and the data flagged to indicate the presence of interferences.

4.3. Physical Interferences. Physical interferences are associated with the transport and nebulization process. Internal standards are used to compensate for these types of interferences.

4.3.1. Internal standards should be added at a level to give approximately 100,000 – 1,000,000 counts of raw signal intensity. The mass of the internal standard should ideally be within 20 amu of the mass of the measured analyte.

4.3.2. Matrix effects will be monitored by comparing the internal standard intensity in the sample to the internal standard intensity of the calibration blank. When performing Method 6020, the internal standard intensities must be between 30% and 120% of the intensities in the calibration blank. When performing Method 200.8, the internal standards must be between 60% and 125% of the calibration blank. If they fall outside this window, a dilution is performed on the sample to correct for matrix effects and the sample re-analyzed.

4.3.3. Memory effects are dependent on the relative concentration differences between samples and/or standards, which are analyzed sequentially. The rinse period between samples must be long enough to eliminate significant memory interference.

4.3.4. Method interferences may be caused by contaminants in solvents, reagents, glassware, and other processing apparatus that lead to discrete artifacts. All of these materials must be demonstrated routinely to be free from interferences under conditions of the analysis by running laboratory method blanks as described in the Quality Control section. All glassware is cleaned per SOP NC-QA-014.Specific selection of reagents may be required to avoid introduction of contaminants.

5. SAFETY

5.1. Employees must abide by the policies and procedures in the Corporate Environmental

Health and Safety Manual and this document.

5.2. Eye protection that protects against splash, laboratory coat, and appropriate gloves must be worn while samples, standards, solvents, and reagents are being handled. Cut-resistant gloves must be worn doing any other task that presents a strong possibility of getting cut. Disposable gloves that have been contaminated must be removed and discarded; other gloves must be cleaned immediately.

5.3. The following is a list of the materials used in this method, which have a serious or significant hazard rating. **NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the Reagents and Standards section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Hydrochloric Acid	Corrosive Poison	5 ppm-Ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Nitric Acid	Corrosive Oxidizer Poison	2 ppm-TWA 4 ppm-STEL	Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison. Inhalation of vapors can cause breathing difficulties and lead to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract. Can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

5.4. The RF Generator produces strong radio frequency waves--most of which are unshielded. People with pacemakers must not go near the instrument while in operation.

5.5. Exposure to chemicals must be maintained **as low as reasonably achievable**. All samples with stickers that read "Caution/Use Hood!" **must** be opened in the hood. Contact the EH&S Coordinator if this is not possible. Solvent and waste containers must be kept closed unless transfers are being made.

5.6. Standards in solution must be diluted in the open laboratory when syringes and the like are utilized.

5.7. The ICP plasma emits strong UV light and is harmful to vision. All analysts must avoid looking directly at the plasma.

5.8. All work must be stopped in the event of a known or potential compromise to the health and safety of a TestAmerica associate. The situation must be reported **immediately** to a laboratory supervisor and the EH&S Coordinator.

6. EQUIPMENT AND SUPPLIES

6.1. Argon gas: High purity grade (99.99%)

6.2. Inductively Coupled Plasma Mass Spectrometer capable of providing resolution, less than or equal to 0.9 AMU at 10% peak height from 6-253 AMU and 1.0AMU at 5% peak height from 6-253 AMU with a data system that allows corrections for isobaric interferences and the application of the internal standard technique.

6.3. A four channel peristaltic pump

6.4. Appropriate water-cooling device

6.5. Calibrated automatic pipettes

6.6. Autosampler with autosampler tubes

6.7. Hydrogen/helium gas mixture with approximate ratio of 7% hydrogen and 93% helium used for CCT mode.

7. REAGENTS AND STANDARDS

7.1. Calibration standards are purchased as custom multi-element mixes or as single element solutions. All standards must be stored in FEP fluorocarbon or previously unused polyethylene or polypropylene bottles. Intermediate standard solutions must be replaced prior to the expiration date provided by the manufacturer. If no expiration date is provided, the intermediate solutions may be used for up to one year and must be replaced sooner if verification from an independent source indicates a problem. Additional information can be found in SOP NC-QA-017.

7.2. Check Calibration Standard (ICV) - A quality control standard similar to the calibration standards and prepared in the same acid matrix. This solution must be made at a concentration near the midpoint of the calibration curve. This standard is composed of analytes from a different source from those used in the calibration of the instrument. See Table VI. Refer to the Standard Logbook for details on standard preparation.

7.3. The tuning solution is purchased as custom multi-element mixes or as single element solutions. All standards must be stored in FEP fluorocarbon or previously unused polyethylene or polypropylene bottles. The solution must be replaced prior to the expiration date provided by the manufacturer. If no expiration date is provided, the intermediate solutions may be used for up to one year.

7.4. Reagent water - ASTM Type I or equivalent for the elements of interest, generated using an ion-exchange water polishing system.

7.5. Rinse Solution -.Carefully dilute 80 ml of concentrated HNO3 to 4.0 L with reagent water.

8. SAMPLE COLLECTION, PRESERVATION, AND STORAGE

8.1. Aqueous samples are preserved with nitric acid to a pH of < 2, and may be stored in plastic or glass. Preservation must be verified prior to analysis.

8.2. Soil samples do not require preservation, but must be stored at 4° ± 2°C until the time of preparation.

8.3. The analytical holding times for metals are six months from the time of collection to analysis.

8.4. Solid and aqueous samples must be digested prior to analysis by the appropriate method.

9. QUALITY CONTROL

9.1. Initial Demonstration of Capability

Table V provides a summary of quality control requirements including type, frequency, acceptance criteria and corrective action. Prior to analysis of any analyte the following requirements must be met.

9.1.1. Instrument Detection Limit (IDL). IDLs can be determined by calculating the average of the standard deviations of the three runs on three non-consecutive days from the analysis of a reagent blank solution with seven consecutive measurements per day. Each measurement must be performed as a separate analytical sample. The IDL must be performed every three months. The IDL is

calculated by multiplying by 3, the average of the standard deviations obtained on three nonconsecutive days.

9.1.1.1. IDL = (3) (s), where s = standard deviation.

9.1.2. Linear Calibration Ranges - Linear calibration ranges are primarily detector limited. The linear range must be determined at instrument setup, and the upper limit must be verified annually or whenever a change in instrument hardware or operating conditions, in the judgement of the analyst, may lower expected ranges. Standards used to determine or verify linear ranges must be analyzed during a routine analytical run. The linear range is the concentration above which sample results cannot be reported.

9.1.2.1. For initial determination of the upper limit of the linear range, determine the signal responses from three different concentration standards across the estimated range. One standard must be at the upper limit of the estimated range. Results must recover within 10% of the expected value for the three standards. The linear range is then set at the concentration of the high standard.

9.1.2.2. For verification of the upper limit of the linear range, the high standard must recover within 10% of its expected value

9.2. Batch Definition

9.2.1. A batch is a group of no greater than 20 samples excluding QC samples (LCS, Method Blank, MS, MSD) which are processed similarly with respect to the procedure. All sample setups must be initiated within a 24-hour period from the initial preparation or extraction and without interruption of the process. All samples within the batch must be treated with the same lots of reagents and the same processes.

9.3. Method Blank

9.3.1. One method blank (MB) must be processed with each preparation batch. The method blank consists of reagent water containing all reagents specific to the method that is carried through the entire analytical procedure, including preparation and analysis. The method blank is used to identify any system and process interferences or contamination of the analytical system that may lead to the reporting of elevated analyte concentrations or false positive data. The method blank must not contain any analyte of interest at, or above, the reporting limit (exception: common laboratory contaminants, see below) or at, or above, 5% of the measured concentration of that analyte in associated samples, whichever is higher (sample result must be a minimum of 20x higher than the blank

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contamination level).

Note: For Ohio VAP samples, all analytes must be less than the reporting limit unless the samples are non-detect.

9.3.2. Corrective Action for Method Blank

9.3.2.1. If the analyte is a common laboratory contaminant (copper, iron, lead, or zinc), the data may be reported with qualifiers if the concentration of the analyte in the method blank is less than two times the RL. Barium, chromium, and manganese may also be considered common laboratory contaminants at ICPMS reporting limits. **Such action must be addressed in the project narrative. This is not applicable for Ohio VAP samples.**

9.3.2.2. Repreparation and re-analysis of all samples associated with an unacceptable method blank is required when reportable concentrations are determined in the samples (see exception noted above).

9.3.2.3. If there is no analyte greater than the RL in the samples associated with an unacceptable method blank, the data may be reported with qualifiers. Such action must be addressed in the project narrative.

9.3.2.4. If the above criteria are not met and re-analysis is not possible, then the sample data must be qualified. This anomaly must be addressed in the project narrative.

9.4. Laboratory Control Sample (LCS)

9.4.1. One LCS from an independent source must be processed with each preparation batch. The LCS must be carried through the entire analytical procedure. The LCS is used to monitor the accuracy of the analytical process. Ongoing monitoring of the LCS results provides evidence that the laboratory is performing the method within acceptable accuracy and precision guidelines. The historical limits for the LCS for each analyte are in the LIMS system. If the LCS exceeds these limits for any analyte, that analyte is judged to be out of control and must be corrected before the analysis can be reported

9.4.2. Corrective Action for LCS

9.4.2.1. If any analyte is outside established control limits, the system is out of control and corrective action must occur.

9.4.2.2. The only exception is if the LCS recoveries are biased high and the

associated sample is ND for the parameter(s) of interest, the batch is acceptable. **This must be addressed in the project narrative.**

9.4.2.3. Corrective action will be repreparation and re-analysis of the batch unless the client agrees that other corrective action is acceptable. **Note:** For Ohio VAP samples, the batch must be redigested if the exception in Section 9.4.2.2 is not applicable.

9.5. Matrix Spike/Matrix Spike Duplicate (MS/MSD)

9.5.1. One MS/MSD pair must be processed for each batch. A matrix spike (MS) is a field sample to which known concentrations of target analytes have been added. A matrix spike duplicate (MSD) is a second aliquot of the same sample (spiked identically as the MS) prepared and analyzed along with the sample and matrix spike. Some client-specific data quality objectives (DQOs) may require the use of sample duplicates in place of, or in addition to, MS/MSDs. The MS/MSD results are used to determine the effect of a matrix on the precision and accuracy of the analytical process. Due to the potential variability of the matrix of each sample, these results may have immediate bearing only on the specific sample spiked. Samples identified as field blanks cannot be used for MS/MSD analysis. The historical spike recovery acceptance limits for each analyte are in the LIMS system. If they are not in control, and all other quality control criteria have been met, then a matrix interference is suspected.

9.5.2. Corrective action for MS/MSDs

9.5.2.1. If the analyte recovery or RPD falls outside the acceptance range, the recovery of that analyte must be in control for the LCS. If the LCS recovery is within limits, then the laboratory operation is in control and the results may be accepted. If the recovery of the LCS is outside limits, corrective action must be taken. Corrective action will include repreparation and re-analysis of the batch.

9.5.2.2. If the native analyte concentration in the MS/MSD exceeds four times the spike level for that analyte, the recovery data is reported as NC (not calculated).

9.5.2.3. If an MS/MSD is not possible due to limited sample volume, then a laboratory control sample duplicate (LCSD) must be analyzed. The RPD of the LCS and LCSD must be compared to the laboratory limits.

9.5.2.4. If client program requirements specify to confirm matrix interferences, repreparation and re-analysis of the MS/MSD may be necessary.

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9.6. Sample Duplicate

9.6.1. A sample duplicate (DU) is a second aliquot of an environmental sample taken from the same sample container, when possible, that is processed with the first aliquot of that sample. That is, sample duplicates are processed as independent samples within the same QC batch. The sample and DU results are compared to determine the effect of the sample matrix on the precision of the analytical process. As with the MS/MSD results, the sample/DU precision results are not necessarily representative of the precision for other samples in the batch.

9.6.2. Sample duplicates may be performed in lieu of, or in addition to, MSDs.

9.7. Control Limits

9.7.1. Control limits are established by the laboratory as described in SOP NC-QA-018.

9.7.2. Laboratory control limits are internally generated and updated periodically unless method specified. Control limits are easily accessible via LIMs (QC Browser program).

9.8. Method Detection Limits (MDLs) and MDL Checks

9.8.1. MDLs and MDL Checks are established by the laboratory as described in SOPs CA-Q-S-006 and NC-QA-021.

9.8.2. MDLs are easily accessible via LIMs (QC Browser program).

9.9. General Corrective Action Requirements. The general requirements for evaluation of QC results and corrective action for failures is described in TestAmerica Policy QA-003. Ohio VAP projects must reference this SOP instead of Policy QA-003 for information on QC Samples.

9.10. Nonconformance and Corrective Action

9.10.1. Any deviations from QC procedures must be documented as a nonconformance with applicable cause and corrective action approved by the facility QA Manager. Procedural deviations are not allowed for Ohio VAP Projects.

10. CALIBRATION AND STANDARDIZATION

10.1. Instrument Startup. Set up the instrument according to manufacturers operating instructions. Allow the instrument to become thermally stable for at least 30 minutes before tuning.

10.2. Instrument Tuning / Mass Calibration / Daily Performance

10.2.1. Daily Performance. Refer to Appendix A for ICPM/MS Instrument Instructions. Instrument manuals are available as needed. Verify instrument performance daily with a solution containing elements representing all of the mass regions of interest. The relative standard deviations must be less than 5% after running the tuning solution a minimum of four times. For Method 200.8, the tuning solution must be analyzed five times with a relative standard deviation less than 5%. Tuning criteria is listed in Table IV.

10.2.2. Check mass calibration and resolution daily.

10.2.2.1. Mass Calibration Check. The mass calibration results must be within 0.1 amu from the true value. If this criterion is not met, the mass calibration must be adjusted before running samples.

10.2.2.2. Mass Resolution Check. The resolution must be verified to be less than 0.9 amu full width at 10% peak height.

10.3. Calibrate the instrument for the analytes of interest according to manufacturer's instructions. Routine calibration and calibration verification levels are shown in Table XI. The calibration should include a minimum of a blank and two standards. For a linear multi-point calibration curve, the correlation coefficient must be >/= 0.995. Report the average of at least two integrations for both calibration and sample analysis. A calibration must be performed daily and each time the instrument is set up. Instrument run may be continued over periods exceeding 24 hours as long as calibration verification, interference check, and internal standard QC criteria are met.

10.4. ICV/CCV/QCS. Calibration accuracy is verified at the beginning of each analytical run by analyzing a second-source initial calibration verification (ICV) standard. A continuing calibration verification (CCV) standard is analyzed at a 10% frequency throughout the run. The ICV must be within 10% of the expected value, or the analysis is terminated. The CCV must be within 10% of the expected value for Method 6020 or 15% of the expected value for Method 200.8. Sample results may only be reported when bracketed by valid CCVs. Table V provides a summary of quality control requirements including frequency, acceptance criteria, and corrective action.

Note: The only exception is if the CCV recoveries are biased high and the associated sample is ND for the parameter(s) of interest, the batch is acceptable. **This must be addressed in the project narrative.**

10.5. RL Verification Standard. An independent standard is analyzed after the ICV to monitor the lab's ability to produce reliable results at RL-level concentrations. There is no set acceptance criteria established for this standard, but generally results should be within 50% of the expected value. Individual program requirements may vary.

10.6. ICB/CCB/CB. The initial calibration blank must be analyzed immediately following the ICV. The continuing calibration blank must be analyzed at a frequency of 10% throughout the remainder of the analytical run. The ICB/CCB must fall within +/- the reporting limit from zero. Table V provides a summary of quality control requirements including frequency, acceptance criteria, and corrective action.

Note: The only exception is if the CCB recoveries are biased high and the associated sample is ND for the parameter(s) of interest, the batch is acceptable. **This must be addressed in the project narrative.**

10.7. Interference Check Solutions (ICSA/ICSAB) ***Method 6020 only***. The interference check solution is prepared with known concentrations of interfering elements so a determination may be made as to the magnitude of the interference on analytes of interest as well as a test of any software corrections. The required elements and their concentrations are listed in Table II. The interference check solutions must be analyzed at the beginning of every analytical run and every 12 hours thereafter. The results of solution "A" and solution "AB" must be monitored for possible interferences.

10.7.1. Control limits of spiked analytes in the ICSA/ICSAB solution are ± 50% of true value. Some projects may require control limits of ± 20% of true value. Control limits of non-spiked analytes are ± two times the reporting limit or less than 1 ug/L.

Note: It may not be possible to obtain absolutely clean ICSA/ICSAB standards. If contamination can be confirmed by another method (ICP/GFAA), acceptance criteria will be applied at that level and the data accepted.

10.8. Internal Standards. The intensities of all internal standards must be monitored throughout the run. The internal standard in the samples must be between 30% and 120% of the intensity of the calibration blank for Method 6020, and between 60% and 125% for Method 200.8. If the sample falls outside of this criteria, perform the following procedures. First, evaluate nearby CCVs and CCBs. If sample internal standard recoveries appear to be related to instrument drift, then rerun affected samples. If sample internal standard recoveries appear to be primarily sample or matrix related, the run will be evaluated to determine if a dilution is needed. If dilutions are needed, then perform appropriate dilutions until the internal standard recoveries are within the method criteria. In no case may sample results be reported with internal standard recoveries greater than 40% higher than recoveries in surrounding CCVs/CCBs. Alternately, the run may be reprocessed with an alternative internal standard that is not in the samples and at an appropriate mass for the masses being reported. See Table I for a list of Internal Standard analytes.

10.9. Serial Dilution ***Method 6020 only.*** One serial five-fold dilution must be analyzed per batch for each matrix. If the analyte concentration is within linear range of the instrument and sufficiently high (generally, a factor of 100 times above the reporting

limit), the serial dilution must agree within 10% of the original analysis. If not, an interference effect must be suspected, the result is flagged, and included in the final report narrative. Samples identified as blanks cannot be used for serial dilution.

10.10. Post-Digestion Spike Addition (PDS) ***Method 6020 only***. If the serial dilution fails to meet the acceptance criteria, a PDS must be performed as follows. An analytical spike added to a portion of a prepared sample, or its dilution, should be recovered within 75 - 125% of the known value. If the PDS fails to meet this criterion, matrix interference is suspected.

11. PROCEDURE

11.1. One-time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, chemistry, sample size, or other parameters. Any variation in procedure must be completely documented using a Nonconformance Memo and is approved by a Technical Specialist and QA Manager. The Nonconformance Memo must be filed in the project file.

11.2. Any unauthorized deviations from this procedure must also be documented as a nonconformance with a cause and corrective action described. Procedural deviations are not allowed for Ohio VAP projects.

11.3. Sample Preparation

11.3.1. Preliminary acid digestion is required for groundwater, aqueous samples, sludges, sediments, and other solid wastes for which total (acid-leachable) elements are requested. See SOPs NC-IP-010 and NC-IP-011 for preparation details.

11.3.2. For DoD work, refer to SOP NC-QA-016 for specific details.

11.4. Sample Analysis

11.4.1. Flush the system with the rinse blank for at least 30 seconds between samples and standards during the analytical run.

11.4.2. Masses which would affect the data quality must be monitored during the analytical run to determine the potential effects of matrix on a given element.

11.4.3. Dilute and re-analyze samples that are more concentrated than the linear range for an analyte or specific isotope of interest. The sample should be diluted to the approximate midrange of the analytical curve, unless the dilution is for internal standard recoveries.

11.4.4. The analytical run sequence must be performed as follows to meet all quality

control criteria:

Warm-up
Verify instrument performance
Calibration blank
Calibration standards
ICV
ICB
RL verification standard
ICSA (6020 Only)
ICSAB (6020 Only)
CCV
CCB
10 Samples
CCV
CCB

11.5. Analytical Documentation

11.5.1. Record all analytical information in the analytical logbook/logsheet, which may be in an electronic format, including the analytical data from standards, blanks, LCSs, MS/MSDs, and any corrective actions or modifications to the method.

11.5.2. All standards are logged into a department standard logbook. All standards are assigned a unique number for identification. Logbooks are reviewed by the supervisor or designee.

11.5.3. Documentation such as all associated instrument printouts (final runs, screens, reruns, QC samples, etc.) and daily calibration data corresponding to all final runs is available for each data file.

11.5.4. Sample results and associated QC are entered into the LIMs after final technical review.

12. DATA ANALYSIS AND CALCULATIONS

Note: The mean of three exposures is used to derive the sample concentrations used in the calculations in this section.

12.1. ICV percent recoveries are calculated according to the equation:

$$\%R = \mathbf{100\ x}\left(\frac{\mathbf{Found\ (ICV)}}{\mathbf{True\ (ICV)}}\right)$$

12.2. CCV percent recoveries are calculated according to the equation:

$$\%R = \mathbf{100\ x}\left(\frac{\mathbf{Found\ (CCV)}}{\mathbf{True\ (CCV)}}\right)$$

12.3. Matrix Spike Recoveries are calculated according to the following equation:

$$\%R = \mathbf{100\ x}\left(\frac{\mathbf{SSR\ -\ SR}}{\mathbf{SA}}\right)$$

Where:
SSR = Spike Sample Result
SR = Sample Result
SA = Spike Added

Note: When sample concentration is less than the method detection limit, use SR = 0 for purposes of calculating % Recovery.

12.4. The relative percent difference (RPD) of sample duplicates are calculated according to the following equation:

$$RPD = \mathbf{100\ x}\left[\frac{\mathbf{(DU1\ -\ DU2)}}{\mathbf{(DU1\ +\ DU2)\ /\ 2}}\right]$$

Where:
DU1 = Sample result
DU2 = Sample duplicate result

12.5. The final concentration for an aqueous sample is calculated as follows:

$$\text{Result (ug/L)} = \frac{\mathbf{(C\ x\ V1\ x\ D)}}{\mathbf{V2}}$$

Where:
C = Concentration from instrument readout, ppb (mean of three exposures)
D = Instrument dilution factor
V1 = Final volume in liters after sample preparation
V2 = Initial volume of sample digested in liters

12.6. The concentration determined in digested solid samples when reported on a wet weight basis is as follows:

$$\text{Result (ug/kg)} = \frac{\mathbf{(C \times V \times D)}}{\mathbf{W}}$$

Where:

C = Concentration from instrument readout, ppb (mean of three exposures)
D = Instrument dilution factor
V = Final volume in liters after sample preparation
W = Weight, in g, of wet sample digested

12.7. Additional equations and calculations are listed in the following SOPs: Calibration Curves (General), CA-Q-S-005, and Selection of Calibration Points, CA-T-P-002.

13. METHOD PERFORMANCE

13.1. Each laboratory must have initial demonstration of performance data on file and corresponding method detection limit files.

13.2. Refer to Table VII for the list of analytes that may be analyzed using this SOP for Methods 6020 and 200.8. Additional analytes may be analyzed if all method-required QC is acceptable.

13.3. Training Qualifications

13.3.1. The Group/Team Leader has the responsibility to ensure that this procedure is performed by an associate who has been properly trained in its use and has the required experience.

14. POLLUTION PREVENTION

14.1. It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage, and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention".

15. WASTE MANAGEMENT

15.1. All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and

the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention".

15.2. Solid materials (gloves, soiled paper products, etc.) are placed in the solid debris container. Do not put liquids in the solid waste container.

15.3. Laboratory personnel assigned to perform hazardous waste disposal procedures must have a working knowledge of the established procedures and practices of TestAmerica. They must have training on the hazardous waste disposal practices upon initial assignment to these tasks followed by an annual refresher training.

15.4. Waste Streams Produced by the Method

15.4.1. Acid waste consisting of sample and rinse solution generated by this method.

15.4.1.1. Aqueous waste can be poured down the drain if the pH is between 5 and 10. Any sample waste generated that is not in this pH range must be collected and disposed of in the acid waste drum located in the metals lab.

16. REFERENCES

16.1. References

16.1.1. Test Methods For Evaluating Solid Waste, EPA SW-846, 3rd Edition, Final Update II, Method 6020: "Inductively Coupled Argon Plasma - Mass Spectrometry", Revision 0, September 1994

16.1.2. Environmental Monitoring Systems Laboratory, EPA Method 200.8, "Determination of Trace Elements in Waters and Wastes by Inductively Coupled Plasma - Mass Spectrometry", Revision 5.4, EMMC version

16.1.3. TestAmerica North Canton Quality Assurance Manual (QAM), current version

16.1.4. TestAmerica Corporate Environmental Health and Safety Manual, CW-E-M-001 (changed from M-E-0001), and TestAmerica North Canton Facility Addendum and Contingency Plan, current version

16.1.5. Revision History

Historical File:		Revision 0: 08/01/95		Revision 4.2: 01/08/04
		Revision 1: 06/06/01		Revision 4.3: 07/28/07
		Revision 3: 03/26/02		Revision 4.4: 07/30/08
		Revision 4: 03/06/03		

		Revision 4.1: 10/01/03		

16.2. Associated SOPs and Policies, current version

16.2.1. QA Policy, QA-003

16.2.2. Acid Digestion of Soils, SW846 Method 3050B, NC-IP-010

16.2.3. Acid Digestion of Aqueous Samples by SW846 and MCAWW 200 Series Methods, NC-IP-011

16.2.4. Glassware Washing, NC-QA-014

16.2.5. Statistical Evaluation of Data and Development of Control Charts, NC-QA-018

16.2.6. Method Detection Limits and Instrument Detection Limits, NC-QA-021 and CA-Q-S-006

16.2.7. Supplemental Practices for DoD Project Work, SOP NC-QA-016

16.2.8. Standards and Reagents, NC-QA-017

16.2.6. Selection of Calibration Points, CA-T-P-002

16.2.7. Calibration Curves (General), CA-Q-S-005

17. MISCELLANEOUS (TABLES, APPENDICES, ETC.)

17.1. Reporting limits

17.1.1. Refer to Table VII for associated reporting limits.

17.1.2. If samples require dilution or smaller volumes than specified in this method, the RL will be elevated.

17.2. Method Deviations

17.2.1. Deviations from Method 6020

17.2.1.1. Commercially available standards are purchased and verified at the laboratory rather than being prepared from the solid material. These verification records are kept in the laboratory.

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17.2.1.2. The results of the calibration blank as well as all other blanks must be less than the reporting limit--not three times the instrument IDL.

17.2.1.3. Milli-Q or Nanopure water is substituted when reagent water is called for. This water is tested to be free of contaminants by the analysis of blanks.

17.2.1.4. Internal standard recoveries may be less than 80% in CCVs and CCBs as long as QC criteria are met. Sample internal standard recoveries may never be greater than 40% higher than recoveries in associated CCVs/CCBs.

17.2.2. Deviations from Method 200.8

17.2.2.1. Commercially available standards are purchased and verified at the laboratory rather than being prepared from the solid material. These verification records are kept in the laboratory.

17.2.2.2. The results of the calibration blank as well as all other blanks must be less than the reporting limit--not three times the instrument IDL.

17.2.2.3. Milli-Q or Nanopure water is substituted when reagent water is called for. This water is tested to be free of contaminants by the analysis of blanks.

17.2.2.4. Resolution criteria of the mass calibration is met if the resolution criteria for Method 6020 is satisfied.

17.2.2.5. The concentration of most analytes in the LCS is 100 µg/L. This is made from a commercially available stock solution and has all analytes at the same level. Verification records for this solution are kept in the laboratory.

17.2.2.6. Results are reported up to the verified linear range--not up to only 90% of the linear range.

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Table I: Recommended Internal Standards

Li
Sc
Y
Rh
In
Tb
Ho
Bi
Ge

Table II: Interference Check Sample Components and Recommended Concentrations

Interference Component	Solution A Concentration (mg/L)	Solution AB Concentration (mg/L)
Al	50	50
Ca	50	50
Fe	50	50
Mg	50	50
Na	50	50
P	50	50
K	50	50
S	50	50
C	100	100
Cl	500	500
Mo	1.0	1.0
Ti	1.0	1.0
As	0.0	0.1
Cd	0.0	0.1
Cr	0.0	0.1
Co	0.0	0.1
Cu	0.0	0.1
Mn	0.0	0.1
Ni	0.0	0.1
Se	0.0	0.1
Ag	0.0	0.1
V	0.0	0.1
Zn	0.0	0.1

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Table III: Tuning Solution

A tuning solution containing elements representing all of the mass regions of interest must be analyzed. Below are two groups of suggested solutions which cover a typical mass calibration range.

Element	Concentration (µg/L)
Mg	10
Rh	10
Pb	10
Li	10
Co	10
In	10
Tl	10

Element	Concentration (µg/L)
Be	10
Mg	10
Co	10
In	10
Pb	10
Ba	10
Ce	10

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Table IV: Suggested Tuning and Response Factor Criteria Minimum Response from Tuning Solution

Minimum Response from Tuning Solution With a Peristaltic Pump Speed of 40 RPM

Be	> 8,000
In	>300,000
Pb	> 100,000
Co	> 100,000
Mg	> 10,000

Suggested Mass Calibration

Be	9.0122
Mg	23.98
Rh	102.91
Pb	207.98
Li	7.016
Co	58.9332
In	114.904
Tl	204.9744

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Table V: Summary of Quality Control Requirements

QC Parameter	Frequency	Acceptance Criteria	Corrective Action
ICV/QCS	Beginning of every analytical run	90 - 110% recovery	Terminate analysis; correct the problem; recalibrate
ICB/CB	Immediately after each ICV	The result must be < RL	Terminate analysis; correct the problem; recalibrate
CCV	Beginning and end of run and every 10 samples	**6020:** 90 - 110% recovery **200.8:** 85-115% recovery	If unacceptable, correct the problem, recalibrate the instrument, reverify calibration, and rerun all samples associated with unacceptable CCVs.
CCB	Immediately following each CCV	The result must be < RL	If unacceptable, correct the problem, recalibrate the instrument, reverify calibration, and rerun all samples associated with unacceptable CCBs.
ICSA (6020 Only)	Beginning and every 12 hours	Monitor for possible interferences	See Section 9.12
ICSAB (6020 Only)	Immediately following each ICSA	Monitor for possible interferences	See Section 9.12
Method Blank/Lab Reagent Blank	One per lot of 20 field samples or fewer	The result must be < RL Sample results greater than 20x the blank concentration or samples for which the contaminant is < RL, do not require redigestion or reanalysis.	Redigest and reanalyze samples See Section 9.3 for additional requirements
Laboratory Control Sample/Lab Fortified Blank	One per lot of 20 field samples or fewer	80-120%, or in-house limits (6020), 85-115% (200.8)	Redigest and reanalyze samples See Section 9.4
Serial Dilution (6020 Only)	One per lot of 20 field samples or fewer	90 – 110% recovery	See section 9.14 for additional requirements
Post-Digestion Spike (6020 Only)	See Section 9.15	75 - 125% recovery	See section 9.15
Matrix Spike/Matrix Spike Duplicate	One per lot of 20 field samples or fewer	Must be within laboratory control limits	See section 9.5 for additional requirements

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Table VI: ICP/MS Calibration and Calibration Verification Checklist
Suggested Levels in µg/L

Element	Calibration		ICV	CCV
	1	2		
Aluminum	500	1000	400	500
Antimony	100	200	80	100
Arsenic	100	200	80	100
Barium	100	200	80	100
Beryllium	100	200	80	100
Boron	100	200	80	100
Cadmium	100	200	40000	50000
Calcium	50000	100000	40000	50000
Chromium	100	200	80	100
Cobalt	25000	50000	80	100
Copper	100	200	80	100
Iron	500	1000	20000	25000
Lead	100	200	80	100
Manganese	50000	10000	400	500
Magnesium	50000	100000	40000	50000
Molybdenum	100	200	80	100
Nickel	100	200	80	100
Potassium	100	200	40000	50000
Selenium	50000	100000	80	100
Sodium	100	200	40000	50000
Silver	100	200	80	100
Strontium	100	200	80	100
Thallium	100	200	80	100
Tin	100	200	80	100
Titanium	100	200	80	100
Tungsten	100	200	80	100
Vanadium	100	200	80	100
Zinc	100	200	80	100

This procedure has been developed for additional elements. Additional elements may be included in the calibration solution at the appropriate levels. Levels may be adjusted to meet specific regulatory or client programs.

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Table VII: Suggested ICP/MS Reporting Limits

Water 6020 and 200.8 Solid 6020 (only)

Compound	RL	Units	RL	Units
Aluminum	50	ug/L	5000	ug/kg
Antimony	2	ug/L	200	ug/kg
Arsenic	5	ug/L	500	ug/kg
Barium	5	ug/L	500	ug/kg
Beryllium	1	ug/L	100	ug/kg
Boron	15	ug/L	1500	ug/kg
Cadmium	1	ug/L	100	ug/kg
Calcium	1000	ug/L	100000	ug/kg
Chromium	2	ug/L	200	ug/kg
Cobalt	1	ug/L	100	ug/kg
Copper	5	ug/L	500	ug/kg
Iron	50	ug/L	5000	ug/kg
Lead	1	ug/L	100	ug/kg
Magnesium	1000	ug/L	100000	ug/kg
Manganese	5	ug/L	500	ug/kg
Molybdenum	2	ug/L	200	ug/kg
Nickel	2	ug/L	100	ug/kg
Potassium	1000	ug/L	100000	ug/kg
Selenium	5	ug/L	500	ug/kg
Silver	1	ug/L	100	ug/kg
Sodium	1000	ug/L	100000	ug/kg
Strontium	10	ug/L	1000	ug/kg
Thallium	1	ug/L	100	ug/kg
Tin	10	ug/L	1000	ug/kg
Titanium	10	ug/L	1000	ug/kg
Tungsten	10	ug/L	1000	ug/kg
Vanadium	5	ug/L	500	ug/kg
Zinc	10	ug/L	1000	ug/kg

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APPENDIX A

OPERATION INSTRUCTION – ICPMS INSTRUCTIONS

Procedure

1. Instrument Set-up

1.1 Configure the X Series with the standard sample introduction equipment, i.e., a glass concentric nebulizer, glass impact bead spray chamber, and a one-piece torch with 1.5mm ID injector tube. A Peltier spray chamber-cooling unit is optional. Ensure the Xi interface cones are fitted. They can be identified as follows:

- Xi Sampler - 1.1 mm orifice, no nipple, no holes around the flat circumference
- Xi Skimmer - Small pointed skimmer mounted in a copper adapter with two screws

1.3 Switch the instrument into the *Operate* state by clicking the *ON* button at the top of the screen. During the automated ignition sequence, the following processes occur:

- Torch purge with argon gas
- RF power match
- Plasma ignition
- Slide valve open
- Electronics on

This process takes about two minutes. Upon successful ignition, the software will display *Operate* in the *Instrument State* bar. In the event of unsuccessful ignition, the software will display an error message and/or place a message in the *Technician Event Log*. Upon unsuccessful ignition, inspect the sample introduction equipment and torch, ensuring a good gas-seal at each connection and ensuring the torch is not misaligned or damaged. If all appears satisfactory, the ignition may be attempted again. If the ignition process consistently fails, contact your local service agent for advice.

1.4 Once the instrument is in the *Operate* state, it must be left for 30 minutes to reach thermal equilibrium prior to starting analytical measurements. The optimization (tuning), performance testing, and instrument set-up calibrations may be performed after 15 minutes. Ensure the peristaltic pump is operated at a default analytical speed of 40%. This is done by clicking on *Instrument*, *Configurations*, *Configuration Editor*, *View Selected Accessories* (network icon), *Peristaltic Pump*, *Connect* (chain icon). Set pump speed to 40% using the slider bar. Click *OK* to close the dialogue box.

1.5 During the initial 15 minutes, the system can be "conditioned" by aspirating the system thoroughly with 5% HCL and 2% nitric acid solution prior to continuing.

1.6 Instrument tuning (optimization) is performed using a 20 µg/L Tune Solution aspirated through the sample uptake tube. Optimization may not be necessary from day to day if the sample introduction system and cones have not been adjusted in any way, and if the instrument fulfills the performance requirements given below. Tune the instrument manually or use *Autotune* while aspirating a 20 µg/L Tune Solution with the sample uptake probe in the tune solution and the internal standard probe in a 5%HCL and 2%HNO3 solution. *Autotune*, using an appropriately defined sequence, is advised. If the CCT gas mode is being used, along with the standard mode for particular elements, both the CCT gas and the standard mode will need to be tuned. See Tables III and IV for additional tuning information.

If the above criteria are not met, do not proceed. Check that the tune solution was prepared as per instructions, and remake, if necessary. If the sensitivity is below the minimum requirement, a new detector plateau may be required, the cones may require cleaning, or the nebulizer or sample uptake lines may have become blocked, or may not be properly clamped on the peristaltic pump. If the CeO/Ce ratio is 0.03, the nebulizer gas flow can be reduced and/or the sampling depth increased, obtaining a corresponding reduction in oxide formation. Recheck the above parameters after taking any remedial action.

1.7 Save the satisfactory instrument settings by clicking on the disk icon on the Tune page. Note this is not necessary if *Autotune* has been used, as the instrument settings are saved automatically (unless manual adjustments have been made after autotuning).

1.8 Perform a cross-calibration (and mass-calibration and detector voltage setup, if required). Note: Retuning may be necessary after performing this routine.

1.9 Aspirate Tune solution and run a *Performance Report* to confirm the mass-calibration, resolution, minimum sensitivity, and maximum cerium oxide requirement, and to verify instrument stability. The performance report acquires five consecutive one-minute runs and calculates the percentage relative standard deviation (RSD) of the five measurements for each isotope. The RSD of the elemental analytes in the performance report must be < 5%. If the performance report fails, check:

- Liquid uptake tubes for kinks or other damage
- Condition and position of the peristaltic pump tubing
- Tightness of the peristaltic pump clamp screws (these must be just tight enough to draw liquid through the tube smoothly)
- Joints of all sample introduction components, ensuring a good seal
- Nebulizer for blockage

- Salt deposition on cones

Remedy the above as necessary, and repeat the test. Note: Retuning may be required if any sample introduction components are adjusted or replaced.

If the measured mass position for each mass in the performance report is not within ± 0.1 amu of the nominal mass position, a new mass calibration must be performed.

2. Sample Analysis

2.1 Open the method template by clicking on *Templates* and then <Test America North Canton Multimode>. The method template will be opened. This contains all the saved analytical parameters, and only the sample list need be amended.

2.2 Go to *Sample List*. This grid contains all the information about calibration, QC, and samples to be analyzed. The calibration and QC concentration information are already stored. Enter all unknown samples into the list in the appropriate order below the existing calibration and QC samples by overwriting the sample label fields. Delete any QC samples that do not apply to the required method. (If sample list changes are to be made permanent to the method, save the method as a *Template* by going to *File*, *Save As Template.* Enter a new name to create an amended method, or use the same name to overwrite the current one.)

2.3 Once all the sample information is added, check that the required autosampler positions have been correctly entered. Amend as necessary. To sequentially renumber positions, add the correct position required for the initiation of the sequence and right mouse click on the first correctly numbered cell. A pop-up menu will appear. Select *Renumber autosampler positions* from this. Ensure all samples have one survey run and three main runs.

2.4 Save the experiment run by clicking on the *File* menu, then *Save As*. Enter the required file name, e.g., 100107, and click *Save.*

2.5 To print the sample list, go to *Reports,* and check the *Sample List* box. Click the refresh icon. The sample list will be displayed in a printable format. Press the print icon.

3. Loading the Autosampler

3.1 Pour the required samples into pre-cleaned 15 mL polypropylene test tubes. To avoid contamination, a small amount of the solution to be analyzed can be poured into the tube and then discarded. This will rinse out any residual contamination.

3.2 Pour blanks, standards, and QCs (positioned in rack 0) into pre-cleaned 50 mL polypropylene tubes. To avoid contamination, a small amount of the solution to be

analyzed can be poured into the tube and then discarded. This will rinse out any residual contamination. Note that 5 % HCL and 2% nitric acid is used as the calibration blank, ICB, and CCB.

3.3 Place the tubes for each sample into the appropriate position in the rack according to the sample list. Note that the autosampler works on a two-dimensional grid position system by rack number (0-4).

4. Initiating Analysis

4.1 Place the sample probe into the autosampler arm and the internal standard probe into the internal standard solution.

4.2 Go to *Instrument*, *Tune*, and click on the accessories dialog icon. Click on *Autosampler*, and then on the chain icon to connect. The autosampler should initialize. Ensure the probe is at the correct height by positioning it so its tip just protrudes through the hole in the bottom of the arm. Click on the *Go to Wash* icon (faucet) to send the probe to the wash station. Ensure the wash solution is being correctly delivered to the wash station via the peristaltic pump at the rear of the autosampler. Allow at least two minutes for the liquid to be delivered to the sample introduction system.

4.3 Click on the experiment to be run. Click the *Queue* icon, then *Append,* and *OK*. The analysis has now been initiated.

4.4 To monitor the progress of the analysis, right-mouse click on the *MS* icon at the bottom-right of the screen and select *Open Service Window* from the pop-up menu. The Service Window hovers over the current application window until moved or closed and displays the current instrument activity. This window is also used **to stop an analysis,** if required. This is done by clicking on the ***XQ* icon**.

4.5 To view results as they are generated, click on the experiment icon and go to the *Results* tab. Click on the *Refresh* button or the refresh icon (green circular arrows on a page) to calculate the results from the data obtained.

4.6 To view calibration plots, click on the *Calibration Data* tab. The calibration for each analyte can be viewed by clicking on the required isotope in the *Analyte* box. Each subsequent set of calibrations (calibration block) can be displayed by selecting the required calibration block from the drop-down combo box, e.g., *FQ Block 1*, *FQ Block 2*, etc. FQ denotes a Fully-Quantitative calibration, and SQ denotes a Semi-Quantitative calibration, i.e., a response curve generated from the *FQ* calibrations. The SQ response curve is used to calculate semi-quantitative concentrations if required.

4.7 To view data, click on the *Numerical Results* tab. The *Analyte Dilution Conc.* tab is a tabular display of the calculated corrected concentrations for each analyte. These values

have been corrected for internal standardization, external drift correction (if used), and dilution (where entered). The *Mass Uncorrected ICPS* tab shows the uncorrected raw data for each measured mass in units of integrated counts per second (ICPS). The *Analyte ICPS* tab shows integrated counts per second data that has been mathematically corrected for blank deduction, internal standardization, drift correction (if used), and dilution (as appropriate). The *Survey* tabs show the data integrated from the survey scan for each sample. Any concentrations displayed in the survey page will be semi-quantitative only.

4.8 To edit the amount of data on screen (filter the results display), click on the filter icon (funnel and lightening). Alter the numerical values or the check boxes to select the required data to display, and click on *OK*. To jump directly to a particular sample of interest, find the sample in the drop-down combo box at the top of the data display, and click on it.

4.9 To display mass-spectra, click on the *Spectra* tab. Display the spectrum for a particular sample by double-clicking on the sample name in the selection box on the left of the screen. Note several spectra may be overlaid by double-clicking on each sample to be displayed. To zoom into a particular area, click the zoom icon (magnifying glass), and click and drag on the spectral display to zoom into the required area. The dashed-lines represent data acquired in the analogue mode of the detector whilst the solid-lines represent pulse-count data. To remove the noise associated with analogue detection at low signal levels, point at the display and right-mouse click to bring up a menu. Go to *View Options*, and then click on *Eliminate Analogue Noise.* To identify a peak, click on it and wait for the options for that mass to be displayed in the box above the spectral display. To fingerprint a spectrum, double-click on the species to fingerprint in the options box. This will overlay the isotopic pattern for the selected species, based on the lowest relative intensity signal for the pattern masses. The spectra may be navigated by using the arrow buttons above the display. Allow the arrow cursor to hover over each button for an on-screen explanation of its function.

5. Post-Analysis Data Processing

5.1 Internal Standards

- Check the internal standard recovery percentage for each internal standard isotope used for every sample. The percentage for each isotope must be within the range 30 - 120% for Method 6020 and 60 – 125% for Method 200.8.

- If above 120%, check that the other internal standard isotopes show similar deviation. If not, this may be due to the presence of the internal standard element in the sample. This is particularly common with the isotopes of Li, Sc, and Y in environmental materials. If this is the case, the affected internal standard isotope may be excluded for the sample affected as follows. Go to the *Sample List*.

- Find the sample affected, and select it in the list by clicking on the box in the left-hand column. Click *Show Advanced*, and go to *Internal Standards*. Click on *New Internal Standard Set*. Select the affected isotope(s) in the *Internal Standards* box on the right. Remove the affected isotope from the *Internal Standards* box by using the left-hand arrow button (<<). Recalculate the results for this sample by going back to *Results*, and clicking on *Refresh*.

- If any internal standard isotope is outside the range 30-120% and all other internal standard isotopes show similar values for that sample, the instrument may have drifted; or the sample may be producing a suppression or enhancement effect. Find the nearest blank following the sample in question and check its internal standard results. If these are similarly reduced or elevated, the instrument has drifted, and the samples must be re-analyzed from the last compliant blank. If the blank does not exhibit similar drift, the sample must be producing a suppression or enhancement effect due to its matrix. In this case, the sample must be re-analyzed after a five-fold (1:5) or a ten-fold (1:10) dilution to reduce the matrix effect.

6. General protocols

6.1 One-time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, chemistry, sample size, or other parameters. Any variation in procedure must be completely documented using a Nonconformance Memo and is approved by a Technical Specialist and QA Manager. The Nonconformance Memo must be filed in the project file.

6.2 Any unauthorized deviations from this procedure must also be documented as a nonconformance with a cause and corrective action described.

6.3 An analytical run will consist of all customer samples and quality control samples analyzed under a daily initial calibration. Each new initial calibration will begin a new analytical run.

6.4 Type in the QC and sample information into the autosampler table.

7. Initial Calibration

7.1 Open a new dataset using the date of analysis as the file name.

7.2 Open the appropriate method if one already exists, or create a new one for the analytes to be quantitated in the run. Solicit the assistance of a senior ICP-MS operator in creating a new method.

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7.3 All masses which could affect data quality should be monitored to determine potential interferences, either simultaneously during an analytical run, or in a separate scan.

7.4 Internal standards are added to all standards and samples by the instrument prior to analysis.

7.5 Use of an existing autosampler table is suggested.

7.6 Calibration consists of a blank and and two calibration standards in accordance with the manufacturer's procedure. Use the average of three integrations for both calibration and sample analyses.

8. The order of analysis for the initial QC samples and calibration should be:

STD1 (Calibration Blank)
STD2 (Calibration Standard)
STD3 (Calibration Standard)
STD4 (Calibration Standard)
ICV
ICB
CRI (Reporting Limit Verification Standard)
ICSA (Interference check solution)
ICSAB (Interference check solution)
CCV
CCB
Prep QC such as LCS or MB, followed by samples (up to 10)
Rinse
CCV
CCB

To continue the analytical run, add an additional ten runs followed by a rinse and CCV/CCB.

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APPENDIX D

Laboratory Method Detection Limit Studies

Analytical Method Information

Analyte	MDL	Reporting Limit	Surrogate %R	Duplicate RPD	Matrix Spike %R	Matrix Spike RPD	Blank Spike / LCS %R	Blank Spike / LCS RPD
8270C Target Compound List in Water (SW846 8270C)								
Preservation: Cool 4°C								
Container: W_1 L Amber Glass		**Amount Required:** 2 x 1L				**Hold Time:** 7 days		
Unpreserved								
Acenaphthene	0.500	2.00 ug/L		24	50 - 120	24	50 - 120	24
Acenaphthylene	0.400	2.00 ug/L		22	53 - 120	22	53 - 120	22
Anthracene	0.200	2.00 ug/L		18	56 - 124	18	63 - 120	18
Benzo (a) anthracene	0.700	2.00 ug/L		20	57 - 122	20	57 - 122	20
Benzo (a) pyrene	0.600	2.00 ug/L		24	46 - 138	24	46 - 138	24
Benzo (b) fluoranthene	0.700	2.00 ug/L		36	45 - 138	36	45 - 138	36
Benzo (g,h,i) perylene	0.500	2.00 ug/L		19	48 - 137	19	48 - 137	19
Benzo (k) fluoranthene	0.800	2.00 ug/L		34	44 - 134	34	44 - 134	34
4-Bromophenyl phenyl ether	3.00	10.0 ug/L		16	50 - 120	16	52 - 120	16
Butyl benzyl phthalate	3.70	10.0 ug/L		19	59 - 134	19	61 - 133	19
Carbazole	0.400	10.0 ug/L		18	53 - 120	18	60 - 120	18
4-Chloro-3-methylphenol	2.80	10.0 ug/L		37	38 - 120	37	49 - 120	37
4-Chloroaniline	4.80	10.0 ug/L		36	35 - 120	36	39 - 120	36
Bis(2-chloroethoxy)methane	2.60	10.0 ug/L		28	41 - 120	28	43 - 120	28
Bis(2-chloroethyl)ether	4.10	10.0 ug/L		19	42 - 120	19	43 - 120	19
Bis(2-chloroisopropyl)ether	3.50	10.0 ug/L		20	45 - 120	20	45 - 120	20
2-Chloronaphthalene	2.90	10.0 ug/L		25	43 - 120	25	43 - 120	25
2-Chlorophenol	2.40	10.0 ug/L		18	40 - 120	18	40 - 120	18
4-Chlorophenyl phenyl ether	2.80	10.0 ug/L		19	56 - 120	19	56 - 120	19
Chrysene	0.500	2.00 ug/L		20	54 - 123	20	54 - 123	20
Dibenz (a,h) anthracene	0.600	2.00 ug/L		18	50 - 136	18	50 - 136	18
Dibenzofuran	0.700	10.0 ug/L		20	55 - 120	20	55 - 120	20
Di-n-butyl phthalate	3.20	10.0 ug/L		19	63 - 120	19	64 - 120	19
1,4-Dichlorobenzene	2.10	10.0 ug/L		33	42 - 120	33	27 - 120	33
1,2-Dichlorobenzene	2.20	10.0 ug/L		33	29 - 120	33	29 - 120	33
1,3-Dichlorobenzene	2.10	10.0 ug/L		34	27 - 120	34	27 - 120	34
3,3-Dichlorobenzidine	6.40	10.0 ug/L		27	10 - 130	27	49 - 120	27
2,4-Dichlorophenol	2.30	10.0 ug/L		21	39 - 120	21	39 - 120	21
Diethyl phthalate	2.80	10.0 ug/L		25	53 - 120	25	53 - 120	25
2,4-Dimethylphenol	2.60	10.0 ug/L		50	11 - 130	50	28 - 123	50
Dimethyl phthalate	2.70	10.0 ug/L		20	59 - 120	20	59 - 120	20
4,6-Dinitro-2-methylphenol	3.30	25.0 ug/L		24	10 - 157	24	35 - 132	24
2,4-Dinitrophenol	3.10	25.0 ug/L		28	10 - 176	28	21 - 145	28
2,6-Dinitrotoluene	3.00	10.0 ug/L		24	62 - 120	24	62 - 123	24
2,4-Dinitrotoluene	3.00	10.0 ug/L		21	61 - 124	21	61 - 124	21
Di-n-octyl phthalate	3.70	10.0 ug/L		21	56 - 141	21	51 - 141	21
Bis(2-ethylhexyl)phthalate	3.30	10.0 ug/L		19	44 - 134	19	54 - 134	19
Fluoranthene	0.700	2.00 ug/L		18	60 - 120	18	62 - 120	18
Fluorene	0.700	2.00 ug/L		21	59 - 120	21	58 - 120	21
Hexachlorobenzene	2.70	10.0 ug/L		19	54 - 120	19	60 - 120	19
Hexachlorobutadiene	2.70	10.0 ug/L		38	24 - 120	38	24 - 120	38
Hexachlorocyclopentadiene	1.90	10.0 ug/L		38	10 - 120	38	19 - 120	38
Hexachloroethane	2.40	10.0 ug/L		40	26 - 120	40	26 - 120	40
Indeno (1,2,3-cd) pyrene	0.500	2.00 ug/L		18	50 - 136	18	50 - 136	18
Isophorone	2.80	10.0 ug/L		34	42 - 120	34	46 - 120	34
2-Methylnaphthalene	0.600	2.00 ug/L		48	34 - 120	48	34 - 120	48

Analytical Method Information

Analyte	MDL	Reporting Limit	Surrogate %R	Duplicate RPD	Matrix Spike %R	Matrix Spike RPD	Blank Spike / LCS %R	Blank Spike / LCS RPD
2-Methylphenol	2.60	10.0 ug/L		19	36 - 120	19	38 - 120	19
Naphthalene	0.500	2.00 ug/L		45	32 - 120	45	32 - 120	45
3/4-Methylphenol	2.30	10.0 ug/L		24	32 - 120	24	34 - 120	24
3-Nitroaniline	2.90	25.0 ug/L		23	38 - 120	23	54 - 120	23
2-Nitroaniline	2.30	25.0 ug/L		25	51 - 126	25	59 - 121	25
4-Nitroaniline	3.40	25.0 ug/L		23	47 - 124	23	55 - 124	23
Nitrobenzene	2.50	10.0 ug/L		34	41 - 120	34	44 - 120	34
4-Nitrophenol	5.30	25.0 ug/L		29	10 - 120	29	10 - 120	29
2-Nitrophenol	3.00	10.0 ug/L		35	35 - 120	35	42 - 120	35
N-Nitrosodiphenylamine	4.00	10.0 ug/L		23	45 - 124	23	59 - 120	23
N-Nitrosodi-n-propylamine	2.70	10.0 ug/L		19	50 - 121	19	50 - 121	19
Pentachlorophenol	2.60	25.0 ug/L		24	46 - 159	24	36 - 143	24
Phenanthrene	0.700	2.00 ug/L		19	53 - 120	19	60 - 120	19
Phenol	1.70	10.0 ug/L		27	10 - 120	27	10 - 120	27
Pyrene	0.600	2.00 ug/L		19	50 - 129	19	57 - 124	19
1,2,4-Trichlorobenzene	2.60	10.0 ug/L		34	27 - 120	34	27 - 120	34
2,4,6-Trichlorophenol	3.00	10.0 ug/L		26	48 - 125	26	48 - 125	26
2,4,5-Trichlorophenol	2.70	25.0 ug/L		26	51 - 125	26	51 - 125	26
Pyridine	2.70	10.0 ug/L		50	10 - 120	50	10 - 120	50
surr: Terphenyl-d14			13 - 120					
surr: 2,4,6-Tribromophenol			29 - 132					
surr: Phenol-d5			10 - 120					
surr: 2-Fluorobiphenyl			29 - 120					
surr: 2-Fluorophenol			10 - 120					
surr: Nitrobenzene-d5			27 - 120					
1,4-Dichlorobenzene-d4								
Naphthalene-d8								
Acenaphthene-d10								
Phenanthrene-d10								
Chrysene-d12								
Perylene-d12								

Analytical Method Information

Analyte	MDL	Reporting Limit	Surrogate %R	Duplicate RPD	Matrix Spike %R	Matrix Spike RPD	Blank Spike / LCS %R	Blank Spike / LCS RPD
8270C Target Compound List in Soil (SW846 8270C)								
Preservation: Cool 4°C								
Container: 8 oz. Glass Jar		**Amount Required:** 90g				**Hold Time:** 14 days		
Acenaphthene	0.0140	0.333 mg/kg		40	42 - 120	40	49 - 120	40
Acenaphthylene	0.0200	0.333 mg/kg		30	32 - 120	30	52 - 120	30
Anthracene	0.00900	0.333 mg/kg		50	10 - 200	50	58 - 120	50
Benzo (a) anthracene	0.0110	0.333 mg/kg		30	41 - 120	30	57 - 120	30
Benzo (a) pyrene	0.00800	0.333 mg/kg		33	33 - 121	33	55 - 120	33
Benzo (b) fluoranthene	0.0380	0.333 mg/kg		42	26 - 137	42	51 - 123	42
Benzo (g,h,i) perylene	0.00900	0.333 mg/kg		32	21 - 124	32	49 - 121	32
Benzo (k) fluoranthene	0.0370	0.333 mg/kg		39	14 - 140	39	42 - 129	39
4-Bromophenyl phenyl ether	0.150	0.333 mg/kg		31	39 - 120	31	49 - 120	31
Butyl benzyl phthalate	0.125	0.333 mg/kg		37	47 - 124	37	59 - 124	37
Carbazole	0.0160	0.333 mg/kg		29	37 - 120	29	54 - 120	29
4-Chloro-3-methylphenol	0.142	0.333 mg/kg		34	38 - 120	34	49 - 120	34
4-Chloroaniline	0.103	0.333 mg/kg		43	20 - 120	43	41 - 120	43
Bis(2-chloroethoxy)methane	0.128	0.333 mg/kg		41	32 - 120	41	37 - 120	41
Bis(2-chloroethyl)ether	0.151	0.333 mg/kg		41	25 - 120	41	29 - 120	41
Bis(2-chloroisopropyl)ether	0.158	0.333 mg/kg		50	23 - 120	50	28 - 120	50
2-Chloronaphthalene	0.150	0.333 mg/kg		34	39 - 120	34	45 - 120	34
2-Chlorophenol	0.149	0.333 mg/kg		45	28 - 120	45	42 - 120	45
4-Chlorophenyl phenyl ether	0.148	0.333 mg/kg		31	43 - 120	31	52 - 120	31
Chrysene	0.0310	0.333 mg/kg		34	28 - 123	34	55 - 120	34
Dibenz (a,h) anthracene	0.0150	0.333 mg/kg		31	25 - 127	31	50 - 123	31
Dibenzofuran	0.0140	0.333 mg/kg		39	40 - 120	39	54 - 120	39
Di-n-butyl phthalate	0.141	0.333 mg/kg		29	32 - 124	29	58 - 120	29
1,4-Dichlorobenzene	0.147	0.333 mg/kg		50	10 - 120	50	15 - 120	50
1,2-Dichlorobenzene	0.154	0.333 mg/kg		50	10 - 120	50	17 - 120	50
1,3-Dichlorobenzene	0.143	0.333 mg/kg		50	10 - 120	50	13 - 120	50
3,3-Dichlorobenzidine	0.0480	0.667 mg/kg		35	13 - 120	35	54 - 120	35
2,4-Dichlorophenol	0.131	0.333 mg/kg		35	33 - 120	35	43 - 120	35
Diethyl phthalate	0.148	0.333 mg/kg		33	34 - 120	33	52 - 120	33
2,4-Dimethylphenol	0.137	0.333 mg/kg		50	29 - 120	50	47 - 120	50
Dimethyl phthalate	0.157	0.333 mg/kg		31	43 - 120	31	55 - 120	31
4,6-Dinitro-2-methylphenol	0.112	0.833 mg/kg		50	10 - 134	50	27 - 134	50
2,4-Dinitrophenol	0.0990	0.833 mg/kg		50	10 - 145	50	15 - 145	50
2,6-Dinitrotoluene	0.150	0.333 mg/kg		34	43 - 120	34	56 - 120	34
2,4-Dinitrotoluene	0.140	0.333 mg/kg		31	42 - 122	31	55 - 122	31
Di-n-octyl phthalate	0.135	0.333 mg/kg		31	34 - 135	31	48 - 131	31
Bis(2-ethylhexyl)phthalate	0.126	0.333 mg/kg		32	40 - 127	32	51 - 127	32
Fluoranthene	0.0110	0.333 mg/kg		35	38 - 120	35	58 - 120	35
Fluorene	0.0200	0.333 mg/kg		37	41 - 120	37	54 - 120	37
Hexachlorobenzene	0.153	0.333 mg/kg		28	44 - 120	28	56 - 120	28
Hexachlorobutadiene	0.174	0.333 mg/kg		50	17 - 120	50	19 - 120	50
Hexachlorocyclopentadiene	0.101	0.333 mg/kg		50	10 - 120	50	11 - 120	50
Hexachloroethane	0.150	0.333 mg/kg		50	10 - 120	50	14 - 120	50
Indeno (1,2,3-cd) pyrene	0.0310	0.333 mg/kg		32	25 - 123	32	50 - 122	32
Isophorone	0.135	0.333 mg/kg		36	32 - 120	36	43 - 120	36
2-Methylnaphthalene	0.0210	0.333 mg/kg		50	11 - 120	50	36 - 120	50

Analytical Method Information

Analyte	MDL	Reporting Limit	Surrogate %R	Duplicate RPD	Matrix Spike %R	Matrix Spike RPD	Blank Spike / LCS %R	Blank Spike / LCS RPD
2-Methylphenol	0.189	0.333 mg/kg		41	41 - 120	41	47 - 120	41
3/4-Methylphenol	0.166	0.333 mg/kg		39	36 - 127	39	53 - 135	39
Naphthalene	0.0140	0.333 mg/kg		42	25 - 120	42	28 - 120	34
3-Nitroaniline	0.273	0.833 mg/kg		35	36 - 120	35	54 - 120	35
2-Nitroaniline	0.121	0.833 mg/kg		28	46 - 120	28	59 - 120	28
4-Nitroaniline	0.252	0.833 mg/kg		36	35 - 121	36	55 - 121	36
Nitrobenzene	0.159	0.333 mg/kg		44	26 - 120	44	30 - 120	44
4-Nitrophenol	0.153	0.833 mg/kg		47	19 - 136	47	44 - 133	47
2-Nitrophenol	0.196	0.333 mg/kg		43	26 - 120	43	36 - 120	43
N-Nitrosodiphenylamine	0.183	0.333 mg/kg		30	43 - 120	30	56 - 120	30
N-Nitrosodi-n-propylamine	0.152	0.333 mg/kg		41	34 - 120	41	45 - 120	41
Pentachlorophenol	0.123	0.833 mg/kg		32	15 - 135	32	42 - 135	32
Phenanthrene	0.0100	0.333 mg/kg		32	37 - 120	32	56 - 120	32
Phenol	0.131	0.333 mg/kg		42	38 - 120	42	45 - 120	42
Pyrene	0.0230	0.333 mg/kg		40	29 - 125	40	56 - 120	40
1,2,4-Trichlorobenzene	0.144	0.333 mg/kg		46	22 - 120	46	22 - 120	46
2,4,6-Trichlorophenol	0.149	0.333 mg/kg		34	32 - 120	34	50 - 120	34
2,4,5-Trichlorophenol	0.148	0.833 mg/kg		33	39 - 120	33	54 - 120	33
Pyridine	0.101	0.667 mg/kg		50	10 - 120	50	26 - 120	50
surr: Terphenyl-d14			18 - 120					
surr: 2,4,6-Tribromophenol			19 - 120					
surr: Phenol-d5			18 - 120					
surr: 2-Fluorobiphenyl			14 - 120					
surr: 2-Fluorophenol			17 - 120					
surr: Nitrobenzene-d5			17 - 120					
1,4-Dichlorobenzene-d4								
Naphthalene-d8								
Acenaphthene-d10								
Phenanthrene-d10								
Chrysene-d12								
Perylene-d12								

Analytical Method Information

Analyte	MDL	Reporting Limit	Surrogate %R	Duplicate RPD	Matrix Spike %R	Matrix Spike RPD	Blank Spike / LCS %R	Blank Spike / LCS RPD
8260B TCL list in Water (SW846 8260B)								
Preservation: pH<2 HCL, Cool 4°C								
Container: VOA Vial HCl		**Amount Required:** 3 x 40 ml				**Hold Time:** 14 days		
				VOA				
Acetone	25.0	50.0 ug/L		31	56 - 150	31	56 - 150	31
Benzene	0.270	1.00 ug/L		12	65 - 151	12	80 - 121	12
Bromochloromethane	0.440	1.00 ug/L		32	64 - 154	32	73 - 137	32
Bromodichloromethane	0.370	1.00 ug/L		13	75 - 138	13	75 - 131	13
Bromoform	0.220	1.00 ug/L		18	55 - 153	18	65 - 140	18
Bromomethane	0.470	1.00 ug/L		50	13 - 176	50	50 - 150	50
2-Butanone	1.70	50.0 ug/L		37	45 - 164	37	70 - 144	37
tert-Butylbenzene	0.200	1.00 ug/L		20	73 - 153	20	76 - 135	20
n-Butylbenzene	0.220	1.00 ug/L		11	67 - 151	11	68 - 140	11
sec-Butylbenzene	0.200	1.00 ug/L		21	68 - 159	21	72 - 140	21
Carbon disulfide	0.290	1.00 ug/L		28	33 - 187	28	74 - 137	28
Chlorobenzene	0.380	1.00 ug/L		11	78 - 136	11	80 - 121	11
Carbon Tetrachloride	0.270	1.00 ug/L		26	64 - 157	26	71 - 137	26
Chlorodibromomethane	0.300	1.00 ug/L		16	64 - 145	16	68 - 137	16
Chloroethane	0.410	1.00 ug/L		35	48 - 159	35	50 - 146	35
Chloroform	0.470	1.00 ug/L		32	72 - 145	32	73 - 131	32
Chloromethane	0.180	1.00 ug/L		34	10 - 194	34	30 - 132	34
Cyclohexane	0.200	5.00 ug/L		13	58 - 151	13	58 - 136	13
1,2-Dibromo-3-chloropropane	0.200	5.00 ug/L		21	49 - 162	21	56 - 145	21
1,2-Dibromoethane (EDB)	0.340	1.00 ug/L		10	70 - 152	10	80 - 135	10
Methylcyclohexane	0.210	5.00 ug/L		13	60 - 156	13	64 - 131	13
1,2-Dichlorobenzene	0.200	1.00 ug/L		11	80 - 136	11	80 - 125	11
1,3-Dichlorobenzene	0.490	1.00 ug/L		18	72 - 146	18	80 - 128	18
1,4-Dichlorobenzene	0.250	1.00 ug/L		10	75 - 135	10	80 - 120	10
Dichlorodifluoromethane	0.140	1.00 ug/L		32	23 - 159	32	30 - 132	32
1,2-Dichloroethane	0.380	1.00 ug/L		25	72 - 137	25	70 - 134	25
1,1-Dichloroethane	0.320	1.00 ug/L		34	64 - 154	34	75 - 125	34
1,1-Dichloroethene	0.360	1.00 ug/L		31	34 - 151	31	73 - 125	31
trans-1,2-Dichloroethene	0.360	1.00 ug/L		32	57 - 157	32	77 - 125	32
cis-1,2-Dichloroethene	0.380	1.00 ug/L		32	57 - 154	32	71 - 132	32
1,1,2-Trifluorotrichloroethane	0.260	1.00 ug/L		17	73 - 136	17	73 - 134	17
1,2-Dichloropropane	0.330	1.00 ug/L		11	71 - 139	11	72 - 120	11
trans-1,3-Dichloropropene	0.160	1.00 ug/L		26	47 - 157	26	62 - 139	26
cis-1,3-Dichloropropene	0.190	1.00 ug/L		35	56 - 156	35	70 - 140	35
Ethylbenzene	0.320	1.00 ug/L		12	68 - 157	12	78 - 133	12
2-Hexanone	2.20	50.0 ug/L		20	57 - 154	20	60 - 150	20
Isopropylbenzene	0.240	1.00 ug/L		15	69 - 139	15	69 - 120	15
p-Isopropyltoluene	0.210	1.00 ug/L		18	69 - 151	18	72 - 134	18
Methyl Acetate	0.280	10.0 ug/L		40	16 - 140	40	43 - 127	40
Methyl tert-Butyl Ether	0.320	1.00 ug/L		32	56 - 152	32	76 - 120	32
Methylene Chloride	0.150	5.00 ug/L		36	71 - 136	36	80 - 133	36
4-Methyl-2-pentanone	1.60	10.0 ug/L		35	62 - 159	35	62 - 146	35
Naphthalene	0.170	5.00 ug/L		30	56 - 161	30	71 - 139	30
n-Propylbenzene	0.220	1.00 ug/L		23	61 - 167	23	70 - 143	23
Styrene	0.220	1.00 ug/L		29	69 - 150	29	80 - 136	29
1,1,2,2-Tetrachloroethane	0.250	1.00 ug/L		28	76 - 141	28	73 - 131	28

Analytical Method Information

Analyte	MDL	Reporting Limit	Surrogate %R	Duplicate RPD	Matrix Spike %R	Matrix Spike RPD	Blank Spike / LCS %R	Blank Spike / LCS RPD
Tetrachloroethene	0.220	1.00 ug/L		16	63 - 155	16	77 - 131	16
Toluene	0.330	1.00 ug/L		35	61 - 153	35	78 - 125	35
1,2,4-Trichlorobenzene	0.240	1.00 ug/L		23	64 - 147	23	74 - 136	23
1,2,3-Trichlorobenzene	0.140	1.00 ug/L		28	57 - 155	28	71 - 138	28
1,1,1-Trichloroethane	0.350	1.00 ug/L		29	78 - 153	29	75 - 137	29
1,1,2-Trichloroethane	0.380	1.00 ug/L		21	74 - 138	21	80 - 123	21
Trichloroethene	0.370	1.00 ug/L		11	74 - 139	11	74 - 139	11
Trichlorofluoromethane	0.300	1.00 ug/L		33	53 - 149	33	60 - 133	33
1,3,5-Trimethylbenzene	0.230	1.00 ug/L		21	67 - 151	21	75 - 134	21
1,2,4-Trimethylbenzene	0.200	1.00 ug/L		20	69 - 150	20	77 - 134	20
Vinyl chloride	0.360	1.00 ug/L		32	53 - 137	32	60 - 122	32
o-Xylene	0.300	1.00 ug/L		27	62 - 167	27	66 - 150	27
m,p-Xylene	0.570	2.00 ug/L		16	69 - 155	16	78 - 132	16
Xylenes, total	0.870	3.00 ug/L		18	68 - 158	18	78 - 134	18
1,2-Dichloroethene (total)	0.740	1.00 ug/L		33	60 - 152	33	78 - 126	33
Tertiary Butyl Alcohol	2.00	20.0 ug/L		30	14 - 200	30	60 - 140	30
surr: 1,2-Dichloroethane-d4			63 - 140					
surr: Dibromofluoromethane			73 - 131					
surr: Toluene-d8			80 - 120					
surr: 4-Bromofluorobenzene			79 - 125					
Fluorobenzene								
Chlorobenzene-d5								
1,4-Dichlorobenzene-d4								

Analytical Method Information

Analyte	MDL	Reporting Limit	Surrogate %R	Duplicate RPD	Matrix Spike %R	Matrix Spike RPD	Blank Spike / LCS %R	Blank Spike / LCS RPD
8260B TCL list in Soil (SW846 8260B)								
Preservation: Cool 4°C								
Container: VOA Vial NaHSo4			**Amount Required:** 100g				**Hold Time:** 14 days	
Acetone	0.0250	0.0500 mg/kg		50	29 - 181	50	60 - 150	50
Acetonitrile	0.0100	0.0200 mg/kg		50	10 - 200	50	10 - 150	50
Benzene	0.00110	0.00200 mg/kg		50	42 - 141	50	78 - 126	50
Bromochloromethane	0.000780	0.00200 mg/kg		50	41 - 146	50	78 - 126	50
Bromodichloromethane	0.000460	0.00200 mg/kg		50	32 - 155	50	75 - 129	50
Bromoform	0.000650	0.00200 mg/kg		43	10 - 155	43	74 - 133	43
Bromomethane	0.000880	0.00200 mg/kg		46	10 - 199	46	50 - 150	46
2-Butanone	0.00710	0.0500 mg/kg		50	38 - 161	50	68 - 149	50
tert-Butylbenzene	0.000840	0.00200 mg/kg		50	11 - 165	50	80 - 129	50
n-Butylbenzene	0.00170	0.00200 mg/kg		50	10 - 183	50	73 - 143	50
sec-Butylbenzene	0.000890	0.00200 mg/kg		50	10 - 170	50	76 - 135	50
Carbon disulfide	0.000670	0.00500 mg/kg		48	50 - 136	48	80 - 132	48
Chlorobenzene	0.000720	0.00200 mg/kg		50	25 - 151	50	80 - 123	50
Carbon Tetrachloride	0.000670	0.00200 mg/kg		44	30 - 159	44	70 - 138	44
Chlorodibromomethane	0.000640	0.00200 mg/kg		48	27 - 150	48	80 - 127	48
Chloroethane	0.00100	0.00500 mg/kg		50	15 - 197	50	55 - 150	50
Chloroform	0.000480	0.00200 mg/kg		50	33 - 148	50	70 - 127	50
Chloromethane	0.00110	0.00200 mg/kg		44	10 - 166	44	36 - 137	44
Cyclohexane	0.000570	0.0100 mg/kg		40	26 - 165	40	67 - 126	40
1,2-Dibromo-3-chloropropane	0.00150	0.00500 mg/kg		45	10 - 167	45	62 - 150	45
1,2-Dibromoethane (EDB)	0.000670	0.00200 mg/kg		45	30 - 155	45	80 - 131	45
Methylcyclohexane	0.00330	0.0100 mg/kg		27	11 - 151	27	74 - 122	27
1,2-Dichlorobenzene	0.000670	0.00200 mg/kg		50	10 - 168	50	80 - 127	50
1,3-Dichlorobenzene	0.00150	0.00200 mg/kg		50	10 - 173	50	80 - 131	50
1,4-Dichlorobenzene	0.000720	0.00200 mg/kg		50	10 - 170	50	80 - 129	50
Dichlorodifluoromethane	0.00140	0.00200 mg/kg		50	10 - 188	50	30 - 150	50
1,2-Dichloroethane	0.000510	0.00200 mg/kg		50	32 - 155	50	70 - 139	50
1,1-Dichloroethane	0.000670	0.00200 mg/kg		50	51 - 135	50	71 - 126	50
1,1-Dichloroethene	0.000670	0.00200 mg/kg		50	46 - 141	50	70 - 125	50
trans-1,2-Dichloroethene	0.000760	0.00200 mg/kg		40	41 - 146	40	73 - 128	40
1,1,2-Trifluorotrichloroethane	0.000760	0.00200 mg/kg		46	30 - 169	46	61 - 134	46
cis-1,2-Dichloroethene	0.000670	0.00200 mg/kg		50	32 - 150	50	75 - 126	50
1,2-Dichloropropane	0.000620	0.00200 mg/kg		50	34 - 139	50	75 - 120	50
trans-1,3-Dichloropropene	0.000550	0.00200 mg/kg		48	24 - 151	48	73 - 128	48
cis-1,3-Dichloropropene	0.000350	0.00200 mg/kg		50	23 - 152	50	74 - 136	50
Ethylbenzene	0.000980	0.00200 mg/kg		50	21 - 165	50	79 - 130	50
2-Hexanone	0.00850	0.0500 mg/kg		50	13 - 174	50	65 - 150	50
Isopropylbenzene	0.000670	0.00200 mg/kg		50	20 - 139	50	65 - 121	50
p-Isopropyltoluene	0.000940	0.00200 mg/kg		50	10 - 164	50	76 - 133	50
Methyl Acetate	0.00160	0.0100 mg/kg		50	10 - 200	50	11 - 150	50
Methyl tert-Butyl Ether	0.000670	0.00200 mg/kg		50	34 - 154	50	70 - 128	50
Methylene Chloride	0.00160	0.0100 mg/kg		50	36 - 163	50	69 - 140	50
4-Methyl-2-pentanone	0.00340	0.0500 mg/kg		45	19 - 176	45	67 - 147	45
Naphthalene	0.00170	0.00500 mg/kg		50	10 - 160	50	72 - 150	50
n-Propylbenzene	0.000860	0.00200 mg/kg		50	16 - 174	50	76 - 133	50
Styrene	0.000740	0.00200 mg/kg		50	10 - 177	50	80 - 140	50

Analytical Method Information

Analyte	MDL	Reporting Limit	Surrogate %R	Duplicate RPD	Matrix Spike %R	Matrix Spike RPD	Blank Spike / LCS %R	Blank Spike / LCS RPD
1,1,2,2-Tetrachloroethane	0.000890	0.00200 mg/kg		45	27 - 163	45	75 - 135	45
Tetrachloroethene	0.000670	0.00200 mg/kg		50	33 - 155	50	76 - 130	50
Toluene	0.000890	0.00200 mg/kg		50	45 - 145	50	76 - 126	50
1,2,4-Trichlorobenzene	0.000670	0.00200 mg/kg		50	10 - 175	50	64 - 150	50
1,2,3-Trichlorobenzene	0.000630	0.00200 mg/kg		50	10 - 182	50	75 - 150	50
1,1,1-Trichloroethane	0.000850	0.00200 mg/kg		41	39 - 148	41	70 - 132	41
1,1,2-Trichloroethane	0.00119	0.00500 mg/kg		50	43 - 145	50	73 - 133	50
Trichloroethene	0.00100	0.00200 mg/kg		50	39 - 150	50	79 - 129	50
Trichlorofluoromethane	0.000670	0.00200 mg/kg		47	25 - 174	47	52 - 148	47
1,3,5-Trimethylbenzene	0.000830	0.00200 mg/kg		50	38 - 148	50	80 - 134	50
1,2,4-Trimethylbenzene	0.00127	0.00200 mg/kg		50	22 - 164	50	80 - 132	50
Vinyl chloride	0.00101	0.00200 mg/kg		39	32 - 163	39	53 - 142	39
o-Xylene	0.00118	0.00200 mg/kg		50	36 - 148	50	80 - 131	50
m,p-Xylene	0.000670	0.00300 mg/kg		50	31 - 159	50	80 - 131	50
Xylenes, total	0.00190	0.00500 mg/kg		50	31 - 159	50	80 - 130	50
1,2-Dichloroethene (total)	0.00143	0.00200 mg/kg		41	38 - 147	41	74 - 126	41
Tertiary Butyl Alcohol	0.0110	0.0500 mg/kg		50	10 - 200	50	41 - 150	50
surr: 1,2-Dichloroethane-d4			67 - 138					
surr: Dibromofluoromethane			75 - 125					
surr: Toluene-d8			76 - 129					
surr: 4-Bromofluorobenzene			67 - 147					
Fluorobenzene								
Chlorobenzene-d5								
1,4-Dichlorobenzene-d4								

Analytical Method Information

Analyte	MDL	Reporting Limit	Surrogate %R	Duplicate RPD	Matrix Spike %R	Matrix Spike RPD	Blank Spike / LCS %R	Blank Spike / LCS RPD
8082 PCBs in Water (SW846 8082)								
Preservation: Cool 4°C								
Container: W_1 L Amber Glass		**Amount Required:** 2000 ml				**Hold Time:** 365 days		
Unpreserved								
PCB-1016	0.132	0.500 ug/L		50	36 - 138	50	63 - 120	50
PCB-1016 [2C]	0.0950	0.500 ug/L		50	36 - 138	50	63 - 120	50
PCB-1221	0.186	0.500 ug/L		50	36 - 138	50	36 - 138	50
PCB-1221 [2C]	0.410	0.500 ug/L		50	36 - 138	50	36 - 138	50
PCB-1232	0.215	0.500 ug/L		50	36 - 138	50	36 - 138	50
PCB-1232 [2C]	0.267	0.500 ug/L		50	36 - 138	50	36 - 138	50
PCB-1242 [2C]	0.232	0.500 ug/L		50	36 - 138	50	37 - 122	50
PCB-1242	0.188	0.500 ug/L		50	36 - 138	50	37 - 122	50
PCB-1248 [2C]	0.107	0.500 ug/L		50	36 - 138	50	21 - 120	50
PCB-1248	0.141	0.500 ug/L		50	36 - 138	50	54 - 120	50
PCB-1254 [2C]	0.123	0.500 ug/L		50	36 - 138	50	58 - 123	50
PCB-1254	0.111	0.500 ug/L		50	36 - 138	50	58 - 123	50
PCB-1260	0.200	0.500 ug/L		50	36 - 138	50	36 - 138	50
PCB-1260 [2C]	0.206	0.500 ug/L		50	36 - 138	50	36 - 138	50
PCB-1262 [2C]	0.118	0.500 ug/L		50	36 - 138	50	36 - 138	50
PCB-1262	0.114	0.500 ug/L		50	36 - 138	50	36 - 138	50
PCB-1268 [2C]	0.132	0.500 ug/L		50	36 - 138	50	36 - 138	50
PCB-1268	0.124	0.500 ug/L		50	36 - 138	50	36 - 138	50
Total PCBs	2.60	3.50 ug/L						
Total PCBs [2C]	2.60	3.50 ug/L						
surr: Tetrachloro-meta-xylene			17 - 142					
surr: Tetrachloro-meta-xylene [2C]			17 - 142					
surr: Decachlorobiphenyl			10 - 149					
surr: Decachlorobiphenyl [2C]			10 - 149					

Analytical Method Information

Analyte	MDL	Reporting Limit	Surrogate %R	Duplicate RPD	Matrix Spike %R	Matrix Spike RPD	Blank Spike / LCS %R	Blank Spike / LCS RPD
8082 PCBs in Soil (SW846 8082)								
Preservation: Cool 4°C								
Container: 4 oz. Glass Jar		**Amount Required:** 100 g				**Hold Time:** 365 days		
PCB-1016 [2C]	0.0140	0.0333 mg/kg		50	20 - 175	50	64 - 122	50
PCB-1016	0.0210	0.0333 mg/kg		50	20 - 175	50	64 - 122	50
PCB-1221	0.0110	0.0333 mg/kg		50	17 - 175	50	44 - 144	50
PCB-1221 [2C]	0.0130	0.0333 mg/kg		50	17 - 175	50	44 - 144	50
PCB-1232 [2C]	0.0220	0.0333 mg/kg		50	17 - 175	50	44 - 144	50
PCB-1232	0.0160	0.0333 mg/kg		50	17 - 175	50	44 - 144	50
PCB-1242	0.0260	0.0333 mg/kg		35	21 - 175	35	45 - 137	35
PCB-1242 [2C]	0.0210	0.0333 mg/kg		35	21 - 175	35	45 - 137	35
PCB-1248	0.0300	0.0333 mg/kg		50	17 - 151	50	44 - 139	50
PCB-1248 [2C]	0.0240	0.0333 mg/kg		50	17 - 151	50	44 - 139	50
PCB-1254 [2C]	0.0160	0.0333 mg/kg		37	32 - 160	37	72 - 137	37
PCB-1254	0.0110	0.0333 mg/kg		37	32 - 160	37	72 - 137	37
PCB-1260 [2C]	0.0310	0.0333 mg/kg		36	51 - 159	36	56 - 150	36
PCB-1260	0.0280	0.0333 mg/kg		36	51 - 159	36	56 - 150	36
PCB-1262	0.0120	0.0333 mg/kg		50	17 - 175	50	44 - 144	50
PCB-1262 [2C]	0.0250	0.0333 mg/kg		50	17 - 175	50	44 - 144	50
PCB-1268	0.0290	0.0333 mg/kg		50	17 - 175	50	44 - 144	50
PCB-1268 [2C]	0.0190	0.0333 mg/kg		50	17 - 175	50	44 - 144	50
Total PCBs	0.110	1.00 mg/kg						
Total PCBs [2C]	0.110	1.00 mg/kg						
surr: Tetrachloro-meta-xylene			19 - 147					
surr: Tetrachloro-meta-xylene [2C]			19 - 147					
surr: Decachlorobiphenyl			20 - 150					
surr: Decachlorobiphenyl [2C]			20 - 150					

Analytical Method Information

Analyte	MDL	Reporting Limit	Surrogate %R	Duplicate RPD	Matrix Spike %R	Matrix Spike RPD	Blank Spike / LCS %R	Blank Spike / LCS RPD
Aluminum Total EPA 6010B in Water (SW846 6010B)								
Preservation: Add HNO3 to pH<2								
Container: R_250 mL Plastic HNO3			**Amount Required:**			**Hold Time:** 180 days		
Aluminum	0.0500	0.100 mg/L		20	75 - 125	20	80 - 120	20
Antimony Total EPA 6010B in Water (SW846 6010B)								
Preservation: Add HNO3 to pH<2								
Container: R_250 mL Plastic HNO3			**Amount Required:**			**Hold Time:** 180 days		
Antimony	0.00590	0.0100 mg/L		20	75 - 125	20	80 - 120	20
Arsenic Total EPA 6010B in Water (SW846 6010B)								
Preservation: Add HNO3 to pH<2								
Container: R_250 mL Plastic HNO3			**Amount Required:**			**Hold Time:** 180 days		
Arsenic	0.00400	0.0100 mg/L		20	75 - 125	20	80 - 120	20
Barium Total EPA 6010B in Water (SW846 6010B)								
Preservation: Add HNO3 to pH<2								
Container: R_250 mL Plastic HNO3			**Amount Required:**			**Hold Time:** 180 days		
Barium	0.00600	0.0100 mg/L		20	75 - 125	20	80 - 120	20
Beryllium Total EPA 6010B in Water (SW846 6010B)								
Preservation: Add HNO3 to pH<2								
Container: R_250 mL Plastic HNO3			**Amount Required:**			**Hold Time:** 180 days		
Beryllium	0.00100	0.00400 mg/L		20	75 - 125	20	80 - 120	20
Cadmium Total EPA 6010B in Water (SW846 6010B)								
Preservation: Add HNO3 to pH<2								
Container: R_250 mL Plastic HNO3			**Amount Required:**			**Hold Time:** 180 days		
Cadmium	0.000600	0.00100 mg/L		20	75 - 125	20	80 - 120	20
Calcium Total SW 6010B in Water (SW846 6010B)								
Preservation: Add HNO3 to pH<2								
Container: R_250 mL Plastic HNO3			**Amount Required:**			**Hold Time:** 180 days		
Calcium	0.350	1.00 mg/L		20	75 - 125	20	80 - 120	20
Chromium Total EPA 6010B in Water (SW846 6010B)								
Preservation: Add HNO3 to pH<2								
Container: R_250 mL Plastic HNO3			**Amount Required:**			**Hold Time:** 180 days		
Chromium	0.00100	0.00500 mg/L		20	75 - 125	20	80 - 120	20
Cobalt Total SW 6010B in Water (SW846 6010B)								
Preservation: Add HNO3 to pH<2								
Container: R_250 mL Plastic HNO3			**Amount Required:**			**Hold Time:** 180 days		
Cobalt	0.00200	0.0200 mg/L		20	75 - 125	20	80 - 120	20
Copper Total EPA 6010B in Water (SW846 6010B)								
Preservation: Add HNO3 to pH<2								
Container: R_250 mL Plastic HNO3			**Amount Required:**			**Hold Time:** 180 days		
Copper	0.00300	0.0100 mg/L		20	75 - 125	20	80 - 120	20
Iron Total EPA 6010B in Water (SW846 6010B)								
Preservation: Add HNO3 to pH<2								
Container: R_250 mL Plastic HNO3			**Amount Required:**			**Hold Time:** 180 days		
Iron	0.0490	0.0500 mg/L		20	75 - 125	20	80 - 120	20

Analytical Method Information

Analyte	MDL	Reporting Limit	Surrogate %R	Duplicate RPD	Matrix Spike %R	Matrix Spike RPD	Blank Spike / LCS %R	Blank Spike / LCS RPD
Lead Total EPA 6010B in Water (SW846 6010B)								
Preservation: Add HNO3 to pH<2								
Container: R_250 mL Plastic HNO3			**Amount Required:**			**Hold Time:** 180 days		
Lead	0.00290	0.00500 mg/L		20	75 - 125	20	80 - 120	20
Magnesium Total EPA 6010B in Water (SW846 6010B)								
Preservation: Add HNO3 to pH<2								
Container: R_250 mL Plastic HNO3			**Amount Required:**			**Hold Time:** 180 days		
Magnesium	0.0500	1.00 mg/L		20	75 - 125	20	80 - 120	20
Manganese Total EPA 6010B in Water (SW846 6010B)								
Preservation: Add HNO3 to pH<2								
Container: R_250 mL Plastic HNO3			**Amount Required:**			**Hold Time:** 180 days		
Manganese	0.00150	0.0150 mg/L		20	75 - 125	20	80 - 120	20
Nickel Total EPA 6010B in Water (SW846 6010B)								
Preservation: Add HNO3 to pH<2								
Container: R_250 mL Plastic HNO3			**Amount Required:**			**Hold Time:** 180 days		
Nickel	0.00200	0.0100 mg/L		20	75 - 125	20	80 - 120	20
Potassium Total EPA 6010B in Water (SW846 6010B)								
Preservation: Add HNO3 to pH<2								
Container: R_250 mL Plastic HNO3			**Amount Required:**			**Hold Time:** 180 days		
Potassium	0.150	1.00 mg/L		20	75 - 125	20	80 - 120	20
Selenium Total EPA 6010B in Water (SW846 6010B)								
Preservation: Add HNO3 to pH<2								
Container: R_250 mL Plastic HNO3			**Amount Required:**			**Hold Time:** 180 days		
Selenium	0.00390	0.0100 mg/L		20	75 - 125	20	80 - 120	20
Silver Total EPA 6010B in Water (SW846 6010B)								
Preservation: Add HNO3 to pH<2								
Container: R_250 mL Plastic HNO3			**Amount Required:**			**Hold Time:** 180 days		
Silver	0.00200	0.00500 mg/L		20	75 - 125	20	80 - 120	20
Sodium Total EPA 6010B in Water (SW846 6010B)								
Preservation: Add HNO3 to pH<2								
Container: R_250 mL Plastic HNO3			**Amount Required:**			**Hold Time:** 180 days		
Sodium	0.200	1.00 mg/L		20	75 - 125	20	80 - 120	20
Thallium Total EPA 6010B in Water (SW846 6010B)								
Preservation: Add HNO3 to pH<2								
Container: R_250 mL Plastic HNO3			**Amount Required:**			**Hold Time:** 180 days		
Thallium	0.00840	0.0100 mg/L		20	75 - 125	20	80 - 120	20
Vanadium Total SW 6010B in Water (SW846 6010B)								
Preservation: Add HNO3 to pH<2								
Container: R_250 mL Plastic HNO3			**Amount Required:**			**Hold Time:** 180 days		
Vanadium	0.00700	0.0200 mg/L		20	75 - 125	20	80 - 120	20
Zinc Total EPA 6010B in Water (SW846 6010B)								
Preservation: Add HNO3 to pH<2								
Container: R_250 mL Plastic HNO3			**Amount Required:**			**Hold Time:** 180 days		
Zinc	0.00800	0.0500 mg/L		20	75 - 125	20	80 - 120	20

Analytical Method Information

Analyte	MDL	Reporting Limit	Surrogate %R	Duplicate RPD	Matrix Spike %R	Matrix Spike RPD	Blank Spike / LCS %R	Blank Spike / LCS RPD
Aluminum Total EPA 6010B in Soil (SW846 6010B)								
Preservation: Cool 4°C								
Container: 4 oz. Glass Jar			**Amount Required:**			**Hold Time:** 180 days		
Aluminum	8.20	10.0 mg/kg		20	75 - 125	20	80 - 120	20
Antimony Total EPA 6010B in Soil (SW846 6010B)								
Preservation: Cool 4°C								
Container: 4 oz. Glass Jar			**Amount Required:**			**Hold Time:** 180 days		
Antimony	0.800	10.0 mg/kg		20	75 - 125	20	80 - 120	20
Arsenic Total EPA 6010B in Soil (SW846 6010B)								
Preservation: Cool 4°C								
Container: 4 oz. Glass Jar			**Amount Required:**			**Hold Time:** 180 days		
Arsenic	0.700	1.00 mg/kg		20	75 - 125	20	80 - 120	20
Barium Total EPA 6010B in Soil (SW846 6010B)								
Preservation: Cool 4°C								
Container: 4 oz. Glass Jar			**Amount Required:**			**Hold Time:** 180 days		
Barium	0.700	2.00 mg/kg		20	75 - 125	20	80 - 120	20
Beryllium Total EPA 6010B in Soil (SW846 6010B)								
Preservation: Cool 4°C								
Container: 4 oz. Glass Jar			**Amount Required:**			**Hold Time:** 180 days		
Beryllium	0.100	1.00 mg/kg		20	75 - 125	20	80 - 120	20
Cadmium Total EPA 6010B in Soil (SW846 6010B)								
Preservation: Cool 4°C								
Container: 4 oz. Glass Jar			**Amount Required:**			**Hold Time:** 180 days		
Cadmium	0.200	1.00 mg/kg		20	75 - 125	20	80 - 120	20
Calcium Total SW 6010B in Soil (SW846 6010B)								
Preservation: Cool 4°C								
Container: 4 oz. Glass Jar			**Amount Required:**			**Hold Time:** 180 days		
Calcium	4.70	10.0 mg/kg		20	75 - 125	20	80 - 120	20
Chromium Total EPA 6010B in Soil (SW846 6010B)								
Preservation: Cool 4°C								
Container: 4 oz. Glass Jar			**Amount Required:**			**Hold Time:** 180 days		
Chromium	0.200	1.00 mg/kg		20	75 - 125	20	80 - 120	20
Cobalt Total SW 6010B in Soil (SW846 6010B)								
Preservation: Cool 4°C								
Container: 4 oz. Glass Jar			**Amount Required:**			**Hold Time:** 180 days		
Cobalt	0.300	1.00 mg/kg		20	75 - 125	20	80 - 120	20
Copper Total EPA 6010B in Soil (SW846 6010B)								
Preservation: Cool 4°C								
Container: 4 oz. Glass Jar			**Amount Required:**			**Hold Time:** 180 days		
Copper	0.500	2.00 mg/kg		20	75 - 125	20	80 - 120	20
Iron Total EPA 6010B in Soil (SW846 6010B)								
Preservation: Cool 4°C								
Container: 4 oz. Glass Jar			**Amount Required:**			**Hold Time:** 180 days		
Iron	5.00	10.0 mg/kg		20	75 - 125	20	80 - 120	20

Analytical Method Information

Analyte	MDL	Reporting Limit	Surrogate %R	Duplicate RPD	Matrix Spike %R	Matrix Spike RPD	Blank Spike / LCS %R	Blank Spike / LCS RPD
Lead Total EPA 6010B in Soil (SW846 6010B)								
Preservation: Cool 4°C								
Container: 4 oz. Glass Jar			**Amount Required:**			**Hold Time:** 180 days		
Lead	0.600	1.00 mg/kg		20	75 - 125	20	80 - 120	20
Magnesium Total EPA 6010B in Soil (SW846 6010B)								
Preservation: Cool 4°C								
Container: 4 oz. Glass Jar			**Amount Required:**			**Hold Time:** 180 days		
Magnesium	3.80	10.0 mg/kg		20	75 - 125	20	80 - 120	20
Manganese Total EPA 6010B in Soil (SW846 6010B)								
Preservation: Cool 4°C								
Container: 4 oz. Glass Jar			**Amount Required:**			**Hold Time:** 180 days		
Manganese	0.200	1.00 mg/kg		20	75 - 125	20	80 - 120	20
Mercury 7471A in Soil (SW846 7471A)								
Preservation: Cool 4°C								
Container: 4 oz. Glass Jar			**Amount Required:**			**Hold Time:** 28 days		
Mercury	0.0077	0.10 mg/kg		20	75 - 125	20	80 - 120	20
Nickel Total EPA 6010B in Soil (SW846 6010B)								
Preservation: Cool 4°C								
Container: 4 oz. Glass Jar			**Amount Required:**			**Hold Time:** 180 days		
Nickel	0.400	1.00 mg/kg		20	75 - 125	20	80 - 120	20
Potassium Total EPA 6010B in Soil (SW846 6010B)								
Preservation: Cool 4°C								
Container: 4 oz. Glass Jar			**Amount Required:**			**Hold Time:** 180 days		
Potassium	50.0	100 mg/kg		20	75 - 125	20	80 - 120	20
Selenium Total EPA 6010B in Soil (SW846 6010B)								
Preservation: Cool 4°C								
Container: 4 oz. Glass Jar			**Amount Required:**			**Hold Time:** 180 days		
Selenium	0.600	2.00 mg/kg		20	75 - 125	20	80 - 120	20
Silver Total EPA 6010B in Soil (SW846 6010B)								
Preservation: Cool 4°C								
Container: 4 oz. Glass Jar			**Amount Required:**			**Hold Time:** 180 days		
Silver	0.200	1.00 mg/kg		20	75 - 125	20	75 - 125	20
Sodium Total EPA 6010B in Soil (SW846 6010B)								
Preservation: Cool 4°C								
Container: 4 oz. Glass Jar			**Amount Required:**			**Hold Time:** 180 days		
Sodium	100	200 mg/kg		20	75 - 125	20	80 - 120	20
Thallium Total EPA 6010B in Soil (SW846 6010B)								
Preservation: Cool 4°C								
Container: 4 oz. Glass Jar			**Amount Required:**			**Hold Time:** 180 days		
Thallium	0.900	2.00 mg/kg		20	75 - 125	20	80 - 120	20
Vanadium Total SW 6010B in Soil (SW846 6010B)								
Preservation: Cool 4°C								
Container: 4 oz. Glass Jar			**Amount Required:**			**Hold Time:** 180 days		
Vanadium	0.900	10.0 mg/kg		20	75 - 125	20	80 - 120	20

Analytical Method Information

Analyte	MDL	Reporting Limit	Surrogate %R	Duplicate RPD	Matrix Spike %R	Matrix Spike RPD	Blank Spike / LCS %R	Blank Spike / LCS RPD
Zinc Total EPA 6010B in Soil (SW846 6010B)								
Preservation: Cool 4°C								
Container: 4 oz. Glass Jar		**Amount Required:**				**Hold Time:** 180 days		
Zinc	0.500	10.0 mg/kg		20	75 - 125	20	80 - 120	20

Analytical Method Information

Analyte	MDL	Reporting Limit	Surrogate %R	Duplicate RPD	Matrix Spike %R	Matrix Spike RPD	Blank Spike / LCS %R	Blank Spike / LCS RPD
Mercury Total 7470A in Water (SW846 7470A)								
Preservation: Add HNO3 to pH<2								
Container: R_250 mL Plastic HNO3			**Amount Required:**			**Hold Time:** 28 days		
Mercury	0.000100	0.000200 mg/L		20	75 - 125	20	80 - 120	20

Analytical Method Information

Analyte	MDL	Reporting Limit	Surrogate %R	Duplicate RPD	Matrix Spike %R	Matrix Spike RPD	Blank Spike / LCS %R	Blank Spike / LCS RPD
Mercury 7471A in Soil (SW846 7471A)								
Preservation: Cool 4°C								
Container: 4 oz. Glass Jar		**Amount Required:**				**Hold Time:** 28 days		
Mercury	0.0077	0.10 mg/kg		20	75 - 125	20	80 - 120	20

Analytical Method Information

Analyte	MDL	Reporting Limit	Surrogate %R	Duplicate RPD	Matrix Spike %R	Matrix Spike RPD	Blank Spike / LCS %R	Blank Spike / LCS RPD
Cyanide Total 9012B in Water (SW846 9012B)								
Preservation: Dechlorinate; NaOH to pH>12								
Container: O_250 mL Plastic NaOH			**Amount Required:** 100 ml			**Hold Time:** 14 days		
Cyanide	0.00300	0.00500 mg/L		46	70 - 130	46	90 - 110	46
Cyanide Total 9012B in Soil (SW846 9012B)								
Preservation: Cool 4°C								
Container: 4 oz. Glass Jar			**Amount Required:** 50 gm			**Hold Time:** 14 days		
Cyanide	1.80	2.00 mg/kg		50	69 - 135	50	80 - 120	50

Target Analyte List: **TestAmerica**
Method: 8260 **North Canton Laboratory**
Matrix: Water 12/1/2010

Compound	RL	Units	MDL	Units
Acetone	10	ug/L	1.1	ug/L
Benzene	1	ug/L	0.13	ug/L
Bromodichloromethane	1	ug/L	0.15	ug/L
Bromoform	1	ug/L	0.64	ug/L
Bromomethane	1	ug/L	0.41	ug/L
2-Butanone	10	ug/L	0.57	ug/L
Carbon disulfide	1	ug/L	0.13	ug/L
Carbon tetrachloride	1	ug/L	0.13	ug/L
Chlorobenzene	1	ug/L	0.15	ug/L
Dibromochloromethane	1	ug/L	0.18	ug/L
Chloroethane	1	ug/L	0.29	ug/L
Chloroform	1	ug/L	0.16	ug/L
Chloromethane	1	ug/L	0.3	ug/L
Cyclohexane	1	ug/L	0.12	ug/L
1,2-Dibromo-3-chloropropane	2	ug/L	0.67	ug/L
1,2-Dibromoethane	1	ug/L	0.24	ug/L
1,2-Dichlorobenzene	1	ug/L	0.13	ug/L
1,3-Dichlorobenzene	1	ug/L	0.14	ug/L
1,4-Dichlorobenzene	1	ug/L	0.13	ug/L
Dichlorodifluoromethane	1	ug/L	0.31	ug/L
1,1-Dichloroethane	1	ug/L	0.15	ug/L
1,2-Dichloroethane	1	ug/L	0.22	ug/L
cis-1,2-Dichloroethene	1	ug/L	0.17	ug/L
trans-1,2-Dichloroethene	1	ug/L	0.19	ug/L
1,1-Dichloroethene	1	ug/L	0.19	ug/L
1,2-Dichloropropane	1	ug/L	0.18	ug/L
cis-1,3-Dichloropropene	1	ug/L	0.14	ug/L
trans-1,3-Dichloropropene	1	ug/L	0.19	ug/L
Ethylbenzene	1	ug/L	0.17	ug/L
2-Hexanone	10	ug/L	0.41	ug/L
Isopropylbenzene	1	ug/L	0.13	ug/L
Methyl acetate	10	ug/L	0.38	ug/L
Methylcyclohexane	1	ug/L	0.13	ug/L
Methylene chloride	1	ug/L	0.33	ug/L
4-Methyl-2-pentanone	10	ug/L	0.32	ug/L
Methyl tert-butyl ether	5	ug/L	0.17	ug/L
Styrene	1	ug/L	0.11	ug/L
1,1,2,2-Tetrachloroethane	1	ug/L	0.18	ug/L
Tetrachloroethene	1	ug/L	0.29	ug/L
Toluene	1	ug/L	0.13	ug/L
1,2,4-Trichlorobenzene	1	ug/L	0.15	ug/L
1,1,1-Trichloroethane	1	ug/L	0.22	ug/L
1,1,2-Trichloroethane	1	ug/L	0.27	ug/L
Trichloroethene	1	ug/L	0.17	ug/L
Trichlorofluoromethane	1	ug/L	0.21	ug/L
1,1,2-Trichloro-1,2,2-trifluoroethane	1	ug/L	0.28	ug/L
Vinyl chloride	1	ug/L	0.22	ug/L
Xylenes (total)	2	ug/L	0.28	ug/L

Target Analyte List: **TestAmerica**
Method: 8260 **North Canton Laboratory**
Matrix: Solid 12/1/2010

Compound	RL	Units	MDL	Units
Acetone	1000	ug/kg	170	ug/kg
Benzene	250	ug/kg	12	ug/kg
Bromodichloromethane	250	ug/kg	9.9	ug/kg
Bromoform	250	ug/kg	19	ug/kg
Bromomethane	250	ug/kg	29	ug/kg
2-Butanone	1000	ug/kg	43	ug/kg
Carbon disulfide	250	ug/kg	12	ug/kg
Carbon tetrachloride	250	ug/kg	6.4	ug/kg
Chlorobenzene	250	ug/kg	6.4	ug/kg
Dibromochloromethane	250	ug/kg	12	ug/kg
Chloroethane	250	ug/kg	61	ug/kg
Chloroform	250	ug/kg	8.8	ug/kg
Chloromethane	250	ug/kg	14	ug/kg
Cyclohexane	500	ug/kg	40	ug/kg
1,2-Dibromo-3-chloropropane	500	ug/kg	50	ug/kg
1,2-Dibromoethane	250	ug/kg	10	ug/kg
1,2-Dichlorobenzene	250	ug/kg	8.6	ug/kg
1,3-Dichlorobenzene	250	ug/kg	4.8	ug/kg
1,4-Dichlorobenzene	250	ug/kg	8	ug/kg
Dichlorodifluoromethane	250	ug/kg	16	ug/kg
1,1-Dichloroethane	250	ug/kg	17	ug/kg
1,2-Dichloroethane	250	ug/kg	10	ug/kg
cis-1,2-Dichloroethene	250	ug/kg	6.9	ug/kg
trans-1,2-Dichloroethene	250	ug/kg	9.2	ug/kg
1,1-Dichloroethene	250	ug/kg	18	ug/kg
1,2-Dichloropropane	250	ug/kg	8.2	ug/kg
cis-1,3-Dichloropropene	250	ug/kg	7.9	ug/kg
trans-1,3-Dichloropropene	250	ug/kg	20	ug/kg
Ethylbenzene	250	ug/kg	5.4	ug/kg
2-Hexanone	1000	ug/kg	20	ug/kg
Isopropylbenzene	250	ug/kg	6.5	ug/kg
Methyl acetate	500	ug/kg	25	ug/kg
Methylcyclohexane	500	ug/kg	12	ug/kg
Methylene chloride	250	ug/kg	77	ug/kg
4-Methyl-2-pentanone	1000	ug/kg	48	ug/kg
Methyl tert-butyl ether	1000	ug/kg	7.1	ug/kg
Styrene	250	ug/kg	5.6	ug/kg
1,1,2,2-Tetrachloroethane	250	ug/kg	8.9	ug/kg
Tetrachloroethene	250	ug/kg	12	ug/kg
Toluene	250	ug/kg	17	ug/kg
1,2,4-Trichlorobenzene	250	ug/kg	7.3	ug/kg
1,1,1-Trichloroethane	250	ug/kg	21	ug/kg
1,1,2-Trichloroethane	250	ug/kg	12	ug/kg
Trichloroethene	250	ug/kg	9.7	ug/kg
Trichlorofluoromethane	250	ug/kg	16	ug/kg
1,1,2-Trichloro-1,2,2-trifluoroethane	250	ug/kg	39	ug/kg
Vinyl chloride	250	ug/kg	18	ug/kg
Xylenes (total)	500	ug/kg	8.1	ug/kg

Target Analyte List: **TestAmerica**
Method: 8260 **North Canton Laboratory**
Matrix: Solid - Low Level 12/1/2010

Compound	RL	Units	MDL	Units
Acetone	20	ug/kg	1.6	ug/kg
Benzene	5	ug/kg	0.14	ug/kg
Bromodichloromethane	5	ug/kg	0.15	ug/kg
Bromoform	5	ug/kg	0.42	ug/kg
Bromomethane	5	ug/kg	0.32	ug/kg
2-Butanone	20	ug/kg	0.87	ug/kg
Carbon disulfide	5	ug/kg	0.36	ug/kg
Carbon tetrachloride	5	ug/kg	0.31	ug/kg
Chlorobenzene	5	ug/kg	0.33	ug/kg
Dibromochloromethane	5	ug/kg	0.31	ug/kg
Chloroethane	5	ug/kg	0.35	ug/kg
Chloroform	5	ug/kg	0.28	ug/kg
Chloromethane	5	ug/kg	0.26	ug/kg
Cyclohexane	10	ug/kg	0.22	ug/kg
1,2-Dibromo-3-chloropropane	10	ug/kg	0.99	ug/kg
1,2-Dibromoethane	5	ug/kg	0.41	ug/kg
1,2-Dichlorobenzene	5	ug/kg	0.41	ug/kg
1,3-Dichlorobenzene	5	ug/kg	0.29	ug/kg
1,4-Dichlorobenzene	5	ug/kg	0.47	ug/kg
Dichlorodifluoromethane	5	ug/kg	0.35	ug/kg
1,1-Dichloroethane	5	ug/kg	0.25	ug/kg
1,2-Dichloroethane	5	ug/kg	0.28	ug/kg
cis-1,2-Dichloroethene	5	ug/kg	0.18	ug/kg
trans-1,2-Dichloroethene	5	ug/kg	0.35	ug/kg
1,1-Dichloroethene	5	ug/kg	0.37	ug/kg
1,2-Dichloropropane	5	ug/kg	0.49	ug/kg
cis-1,3-Dichloropropene	5	ug/kg	0.18	ug/kg
trans-1,3-Dichloropropene	5	ug/kg	0.32	ug/kg
Ethylbenzene	5	ug/kg	0.42	ug/kg
2-Hexanone	20	ug/kg	0.6	ug/kg
Isopropylbenzene	5	ug/kg	0.37	ug/kg
Methyl acetate	10	ug/kg	0.64	ug/kg
Methylcyclohexane	10	ug/kg	0.24	ug/kg
Methylene chloride	5	ug/kg	1	ug/kg
4-Methyl-2-pentanone	20	ug/kg	0.68	ug/kg
Methyl tert-butyl ether	20	ug/kg	0.25	ug/kg
Styrene	5	ug/kg	0.27	ug/kg
1,1,2,2-Tetrachloroethane	5	ug/kg	0.23	ug/kg
Tetrachloroethene	5	ug/kg	0.41	ug/kg
Toluene	5	ug/kg	0.32	ug/kg
1,2,4-Trichlorobenzene	5	ug/kg	0.58	ug/kg
1,1,1-Trichloroethane	5	ug/kg	0.26	ug/kg
1,1,2-Trichloroethane	5	ug/kg	0.63	ug/kg
Trichloroethene	5	ug/kg	0.29	ug/kg
Trichlorofluoromethane	5	ug/kg	0.25	ug/kg
1,1,2-Trichloro-1,2,2-trifluoroethane	5	ug/kg	0.44	ug/kg
Vinyl chloride	5	ug/kg	0.27	ug/kg
Xylenes (total)	10	ug/kg	0.83	ug/kg

Target Analyte List: **TestAmerica**
Method: 8270 **North Canton Laboratory**
Matrix: Water 12/1/2010

Compound	RL	Units	MDL	Units
Acenaphthene	10	ug/L	0.1	ug/L
Acenaphthylene	10	ug/L	0.1	ug/L
Acetophenone	10	ug/L	0.34	ug/L
Anthracene	10	ug/L	0.1	ug/L
Atrazine	10	ug/L	0.34	ug/L
Benzaldehyde	10	ug/L	0.39	ug/L
Benzo(a)anthracene	10	ug/L	0.1	ug/L
Benzo(b)fluoranthene	10	ug/L	0.1	ug/L
Benzo(k)fluoranthene	10	ug/L	0.1	ug/L
Benzo(ghi)perylene	10	ug/L	0.1	ug/L
Benzo(a)pyrene	10	ug/L	0.1	ug/L
1,1'-Biphenyl	10	ug/L	0.8	ug/L
bis(2-Chloroethoxy)methane	10	ug/L	0.32	ug/L
bis(2-Chloroethyl) ether	10	ug/L	0.1	ug/L
bis(2-Ethylhexyl) phthalate	10	ug/L	0.8	ug/L
4-Bromophenyl phenyl ether	10	ug/L	0.8	ug/L
Butyl benzyl phthalate	10	ug/L	0.8	ug/L
Caprolactam	10	ug/L	0.8	ug/L
Carbazole	10	ug/L	0.28	ug/L
4-Chloroaniline	10	ug/L	0.8	ug/L
4-Chloro-3-methylphenol	10	ug/L	0.8	ug/L
2-Chloronaphthalene	10	ug/L	0.1	ug/L
2-Chlorophenol	10	ug/L	0.29	ug/L
4-Chlorophenyl phenyl ether	10	ug/L	0.3	ug/L
Chrysene	10	ug/L	0.1	ug/L
Dibenz(a,h)anthracene	10	ug/L	0.1	ug/L
Dibenzofuran	10	ug/L	0.1	ug/L
Di-n-butyl phthalate	10	ug/L	0.67	ug/L
3,3'-Dichlorobenzidine	50	ug/L	0.37	ug/L
2,4-Dichlorophenol	10	ug/L	0.8	ug/L
Diethyl phthalate	10	ug/L	0.6	ug/L
2,4-Dimethylphenol	10	ug/L	0.8	ug/L
Dimethyl phthalate	10	ug/L	0.29	ug/L
4,6-Dinitro-2-methylphenol	50	ug/L	2.4	ug/L
2,4-Dinitrophenol	50	ug/L	2.4	ug/L
2,4-Dinitrotoluene	10	ug/L	0.27	ug/L
2,6-Dinitrotoluene	10	ug/L	0.8	ug/L
Di-n-octyl phthalate	10	ug/L	0.8	ug/L
Fluoranthene	10	ug/L	0.1	ug/L
Fluorene	10	ug/L	0.1	ug/L
Hexachlorobenzene	10	ug/L	0.1	ug/L
Hexachlorobutadiene	10	ug/L	0.27	ug/L
Hexachlorocyclopentadiene	10	ug/L	0.8	ug/L
Hexachloroethane	10	ug/L	0.8	ug/L
Indeno(1,2,3-cd)pyrene	10	ug/L	0.1	ug/L
Isophorone	10	ug/L	0.27	ug/L

Target Analyte List: **TestAmerica**
Method: 8270 **North Canton Laboratory**
Matrix: Water 12/1/2010

(Continued)

2-Methylnaphthalene	10	ug/L	0.1	ug/L
2-Methylphenol	10	ug/L	0.8	ug/L
4-Methylphenol	10	ug/L	0.8	ug/L
Naphthalene	10	ug/L	0.1	ug/L
2-Nitroaniline	50	ug/L	0.8	ug/L
3-Nitroaniline	50	ug/L	0.28	ug/L
4-Nitroaniline	50	ug/L	0.8	ug/L
Nitrobenzene	10	ug/L	0.04	ug/L
2-Nitrophenol	10	ug/L	0.28	ug/L
4-Nitrophenol	50	ug/L	2.4	ug/L
N-Nitrosodiphenylamine	10	ug/L	0.31	ug/L
N-Nitrosodi-n-propylamine	10	ug/L	0.8	ug/L
2,2'-oxybis(1-Chloropropane)	10	ug/L	0.4	ug/L
Pentachlorophenol	10	ug/L	2.4	ug/L
Phenanthrene	10	ug/L	0.1	ug/L
Phenol	10	ug/L	0.6	ug/L
Pyrene	10	ug/L	0.1	ug/L
2,4,5-Trichlorophenol	10	ug/L	0.3	ug/L
2,4,6-Trichlorophenol	10	ug/L	0.8	ug/L

Target Analyte List: **TestAmerica**
Method: 8270 **North Canton Laboratory**
Matrix: Solid (Sonication) 12/1/2010

Compound	RL	Units	MDL	Units
Acenaphthene	330	ug/kg	3.3	ug/kg
Acenaphthylene	330	ug/kg	3.3	ug/kg
Acetophenone	66.6	ug/kg	9.2	ug/kg
Anthracene	330	ug/kg	3.3	ug/kg
Atrazine	330	ug/kg	9.1	ug/kg
Benzaldehyde	330	ug/kg	12	ug/kg
Benzo(a)anthracene	330	ug/kg	3.3	ug/kg
Benzo(b)fluoranthene	330	ug/kg	3.3	ug/kg
Benzo(k)fluoranthene	330	ug/kg	3.3	ug/kg
Benzo(ghi)perylene	330	ug/kg	3.3	ug/kg
Benzo(a)pyrene	330	ug/kg	3.3	ug/kg
1,1'-Biphenyl	330	ug/kg	27	ug/kg
bis(2-Chloroethoxy)methane	330	ug/kg	22	ug/kg
bis(2-Chloroethyl) ether	330	ug/kg	2	ug/kg
bis(2-Ethylhexyl) phthalate	330	ug/kg	19	ug/kg
4-Bromophenyl phenyl ether	330	ug/kg	13	ug/kg
Butyl benzyl phthalate	330	ug/kg	10	ug/kg
Caprolactam	330	ug/kg	37	ug/kg
Carbazole	330	ug/kg	27	ug/kg
4-Chloroaniline	330	ug/kg	17	ug/kg
4-Chloro-3-methylphenol	330	ug/kg	21	ug/kg
2-Chloronaphthalene	330	ug/kg	3.3	ug/kg
2-Chlorophenol	330	ug/kg	27	ug/kg
4-Chlorophenyl phenyl ether	330	ug/kg	13	ug/kg
Chrysene	330	ug/kg	1.1	ug/kg
Dibenz(a,h)anthracene	330	ug/kg	3.3	ug/kg
Dibenzofuran	330	ug/kg	3.3	ug/kg
Di-n-butyl phthalate	330	ug/kg	15	ug/kg
3,3'-Dichlorobenzidine	1600	ug/kg	18	ug/kg
2,4-Dichlorophenol	330	ug/kg	20	ug/kg
Diethyl phthalate	330	ug/kg	16	ug/kg
2,4-Dimethylphenol	330	ug/kg	20	ug/kg
Dimethyl phthalate	330	ug/kg	17	ug/kg
4,6-Dinitro-2-methylphenol	1600	ug/kg	80	ug/kg
2,4-Dinitrophenol	1600	ug/kg	80	ug/kg
2,4-Dinitrotoluene	330	ug/kg	27	ug/kg
2,6-Dinitrotoluene	330	ug/kg	21	ug/kg
Di-n-octyl phthalate	330	ug/kg	27	ug/kg
Fluoranthene	330	ug/kg	3.3	ug/kg
Fluorene	330	ug/kg	3.3	ug/kg
Hexachlorobenzene	330	ug/kg	2.1	ug/kg
Hexachlorobutadiene	330	ug/kg	27	ug/kg
Hexachlorocyclopentadiene	1600	ug/kg	27	ug/kg
Hexachloroethane	330	ug/kg	9	ug/kg
Indeno(1,2,3-cd)pyrene	330	ug/kg	3.3	ug/kg
Isophorone	330	ug/kg	13	ug/kg

Target Analyte List: **TestAmerica**
Method: 8270 **North Canton Laboratory**
Matrix: Solid (Sonication) 12/1/2010

(Continued)

2-Methylnaphthalene	330	ug/kg	3.3	ug/kg
2-Methylphenol	330	ug/kg	80	ug/kg
4-Methylphenol	330	ug/kg	80	ug/kg
Naphthalene	330	ug/kg	3.3	ug/kg
2-Nitroaniline	1600	ug/kg	9.1	ug/kg
3-Nitroaniline	1600	ug/kg	16	ug/kg
4-Nitroaniline	1600	ug/kg	26	ug/kg
Nitrobenzene	330	ug/kg	2.2	ug/kg
2-Nitrophenol	330	ug/kg	27	ug/kg
4-Nitrophenol	1600	ug/kg	80	ug/kg
N-Nitrosodiphenylamine	330	ug/kg	21	ug/kg
N-Nitrosodi-n-propylamine	330	ug/kg	27	ug/kg
2,2'-oxybis(1-Chloropropane)	330	ug/kg	9.5	ug/kg
Pentachlorophenol	330	ug/kg	80	ug/kg
Phenanthrene	330	ug/kg	3.3	ug/kg
Phenol	330	ug/kg	27	ug/kg
Pyrene	330	ug/kg	3.3	ug/kg
2,4,5-Trichlorophenol	330	ug/kg	25	ug/kg
2,4,6-Trichlorophenol	330	ug/kg	80	ug/kg

Target Analyte List: **TestAmerica**
Method: 8270 **North Canton Laboratory**
Matrix: Solid (Soxhlet) 12/1/2010

Compound	RL	Units	MDL	Units
Acenaphthene	330	ug/kg	3.3	ug/kg
Acenaphthylene	330	ug/kg	3.3	ug/kg
Anthracene	330	ug/kg	3.3	ug/kg
Benz(a)anthracene	330	ug/kg	3.3	ug/kg
Benzo(b)fluoranthene	330	ug/kg	3.3	ug/kg
Benzo(k)fluoranthene	330	ug/kg	3.3	ug/kg
Benzo(ghi)perylene	330	ug/kg	3.3	ug/kg
Benzo(a)pyrene	330	ug/kg	3.3	ug/kg
bis(2-Chloroethoxy)methane	330	ug/kg	22	ug/kg
bis(2-Chloroethyl) ether	330	ug/kg	2	ug/kg
bis(2-Chloroisopropyl) ether	330	ug/kg	9.5	ug/kg
bis(2-Ethylhexyl) phthalate	330	ug/kg	19	ug/kg
4-Bromophenyl phenyl ether	330	ug/kg	13	ug/kg
Butyl benzyl phthalate	330	ug/kg	10	ug/kg
4-Chloroaniline	330	ug/kg	17	ug/kg
4-Chloro-3-methylphenol	330	ug/kg	21	ug/kg
2-Chloronaphthalene	330	ug/kg	3.3	ug/kg
2-Chlorophenol	330	ug/kg	27	ug/kg
4-Chlorophenyl phenyl ether	330	ug/kg	13	ug/kg
Chrysene	330	ug/kg	1.1	ug/kg
Dibenz(a,h)anthracene	330	ug/kg	3.3	ug/kg
Dibenzofuran	330	ug/kg	3.3	ug/kg
Di-n-butyl phthalate	330	ug/kg	15	ug/kg
1,2-Dichlorobenzene	330	ug/kg	9.7	ug/kg
1,3-Dichlorobenzene	330	ug/kg	11	ug/kg
1,4-Dichlorobenzene	330	ug/kg	20	ug/kg
3,3'-Dichlorobenzidine	1600	ug/kg	18	ug/kg
2,4-Dichlorophenol	330	ug/kg	20	ug/kg
Diethyl phthalate	330	ug/kg	16	ug/kg
2,4-Dimethylphenol	330	ug/kg	20	ug/kg
Dimethyl phthalate	330	ug/kg	17	ug/kg
4,6-Dinitro-2-methylphenol	1600	ug/kg	80	ug/kg
2,4-Dinitrophenol	1600	ug/kg	80	ug/kg
2,4-Dinitrotoluene	330	ug/kg	27	ug/kg
2,6-Dinitrotoluene	330	ug/kg	21	ug/kg
Di-n-octyl phthalate	330	ug/kg	27	ug/kg
Fluoranthene	330	ug/kg	3.3	ug/kg
Fluorene	330	ug/kg	3.3	ug/kg
Hexachlorobenzene	330	ug/kg	2.1	ug/kg
Hexachlorobutadiene	330	ug/kg	27	ug/kg
Hexachlorocyclopentadiene	1600	ug/kg	27	ug/kg
Hexachloroethane	330	ug/kg	9	ug/kg
Indeno(1,2,3-cd)pyrene	330	ug/kg	3.3	ug/kg
Isophorone	330	ug/kg	13	ug/kg
2-Methylnaphthalene	330	ug/kg	3.3	ug/kg
2-Methylphenol	330	ug/kg	80	ug/kg

Target Analyte List: **TestAmerica**
Method: 8270 **North Canton Laboratory**
Matrix: Solid (Soxhlet) 12/1/2010

(Continued)

4-Methylphenol	330	ug/kg	80	ug/kg
Naphthalene	330	ug/kg	3.3	ug/kg
2-Nitroaniline	1600	ug/kg	9.1	ug/kg
3-Nitroaniline	1600	ug/kg	16	ug/kg
4-Nitroaniline	1600	ug/kg	26	ug/kg
Nitrobenzene	330	ug/kg	2.2	ug/kg
2-Nitrophenol	330	ug/kg	27	ug/kg
4-Nitrophenol	1600	ug/kg	80	ug/kg
N-Nitrosodiphenylamine	330	ug/kg	21	ug/kg
N-Nitrosodi-n-propylamine	330	ug/kg	27	ug/kg
Pentachlorophenol	330	ug/kg	80	ug/kg
Phenanthrene	330	ug/kg	3.3	ug/kg
Phenol	330	ug/kg	27	ug/kg
Pyrene	330	ug/kg	3.3	ug/kg
1,2,4-Trichlorobenzene	330	ug/kg	27	ug/kg
2,4,5-Trichlorophenol	330	ug/kg	25	ug/kg
2,4,6-Trichlorophenol	330	ug/kg	80	ug/kg

Target Analyte List: **TestAmerica**
Method: 6010 Trace **North Canton Laboratory**
Matrix: Water 12/1/2010

Compound	RL	Units	MDL	Units
Arsenic	0.01	mg/L	3.2	ug/L
Lead	0.003	mg/L	1.9	ug/L
Selenium	0.005	mg/L	4.1	ug/L
Thallium	0.01	mg/L	4.7	ug/L

Method: 6010
Matrix: Water

Compound	RL	Units	MDL	Units
Aluminum	0.2	mg/L	97	ug/L
Antimony	0.06	mg/L	1.8	ug/L
Barium	0.2	mg/L	0.67	ug/L
Beryllium	0.005	mg/L	0.46	ug/L
Cadmium	0.005	mg/L	0.66	ug/L
Calcium	5	mg/L	130	ug/L
Chromium	0.01	mg/L	2.2	ug/L
Cobalt	0.05	mg/L	1.7	ug/L
Copper	0.025	mg/L	4.5	ug/L
Iron	0.1	mg/L	81	ug/L
Magnesium	5	mg/L	34	ug/L
Manganese	0.015	mg/L	0.41	ug/L
Nickel	0.04	mg/L	3.2	ug/L
Potassium	5	mg/L	72	ug/L
Silver	0.01	mg/L	2.2	ug/L
Sodium	5	mg/L	590	ug/L
Vanadium	0.05	mg/L	0.64	ug/L
Zinc	0.05	mg/L	5	ug/L

Target Analyte List: **TestAmerica**
Method: 6020 **North Canton Laboratory**
Matrix: Water 12/1/2010

Aluminum	50	ug/L	19	ug/L
Antimony	2	ug/L	0.13	ug/L
Arsenic	5	ug/L	0.4	ug/L
Barium	1	ug/L	0.19	ug/L
Beryllium	1	ug/L	0.2	ug/L
Cadmium	1	ug/L	0.13	ug/L
Chromium	2	ug/L	0.71	ug/L
Cobalt	1	ug/L	0.058	ug/L
Copper	2	ug/L	0.29	ug/L
Iron	50	ug/L	26	ug/L
Lead	1	ug/L	0.18	ug/L
Manganese	1	ug/L	0.83	ug/L
Molybdenum	2	ug/L	0.093	ug/L
Nickel	2	ug/L	0.2	ug/L
Selenium	5	ug/L	1.2	ug/L
Silver	1	ug/L	0.08	ug/L
Thallium	1	ug/L	0.14	ug/L
Tin	10	ug/L	0.31	ug/L
Vanadium	20	ug/L	0.44	ug/L
Zinc	20	ug/L	2.3	ug/L

Target Analyte List: **TestAmerica**
Method: 6010 **North Canton Laboratory**
Matrix: Solid Trace 12/1/2010

Compound	RL	Units	MDL	Units
Arsenic	1	mg/kg	0.3	mg/kg
Lead	0.3	mg/kg	0.19	mg/kg
Selenium	0.5	mg/kg	0.45	mg/kg
Thallium	1	mg/kg	0.55	mg/kg

Method: 6010
Matrix: Solid

Compound	RL	Units	MDL	Units
Aluminum	20	mg/kg	9.6	mg/kg
Antimony	6	mg/kg	0.39	mg/kg
Barium	20	mg/kg	0.071	mg/kg
Beryllium	0.5	mg/kg	0.043	mg/kg
Cadmium	0.5	mg/kg	0.036	mg/kg
Calcium	500	mg/kg	16	mg/kg
Chromium	1	mg/kg	0.2	mg/kg
Cobalt	5	mg/kg	0.16	mg/kg
Copper	2.5	mg/kg	0.74	mg/kg
Iron	10	mg/kg	4.9	mg/kg
Magnesium	500	mg/kg	5.1	mg/kg
Manganese	1.5	mg/kg	0.074	mg/kg
Nickel	4	mg/kg	0.27	mg/kg
Potassium	500	mg/kg	6.2	mg/kg
Silver	1	mg/kg	0.1	mg/kg
Sodium	500	mg/kg	66	mg/kg
Vanadium	5	mg/kg	0.12	mg/kg
Zinc	5	mg/kg	1	mg/kg

Target Analyte List: **TestAmerica**
Method: 6020 **North Canton Laboratory**
Matrix: Solid 12/1/2010

Compound	RL	Units	MDL	Units
Aluminum	5	mg/kg	1.3	mg/kg
Antimony	0.2	mg/kg	0.024	mg/kg
Arsenic	0.5	mg/kg	0.062	mg/kg
Barium	0.1	mg/kg	0.046	mg/kg
Beryllium	0.1	mg/kg	0.047	mg/kg
Cadmium	0.1	mg/kg	0.0078	mg/kg
Chromium	0.2	mg/kg	0.04	mg/kg
Cobalt	0.1	mg/kg	0.0038	mg/kg
Copper	0.2	mg/kg	0.043	mg/kg
Iron	5	mg/kg	1	mg/kg
Lead	0.1	mg/kg	0.013	mg/kg
Manganese	0.1	mg/kg	0.047	mg/kg
Molybdenum	0.2	mg/kg	0.063	mg/kg
Nickel	0.1	mg/kg	0.021	mg/kg
Selenium	0.5	mg/kg	0.09	mg/kg
Silver	0.1	mg/kg	0.016	mg/kg
Thallium	0.1	mg/kg	0.013	mg/kg
Tin	1	mg/kg	0.094	mg/kg
Vanadium	0.5	mg/kg	0.041	mg/kg
Zinc	2	mg/kg	0.2	mg/kg

Target Analyte List: **TestAmerica**
Method: 9012 **North Canton Laboratory**
12/1/2010

Matrix: Water

Compound	RL	Units	MDL	Units
Total Cyanide	0.01	mg/L	0.005	mg/L

Matrix: Solid

Compound	RL	Units	MDL	Units
Total Cyanide	0.5	mg/kg	0.1	mg/kg

Target Analyte List: **TestAmerica**
Method: 8082 **North Canton Laboratory**
12/1/2010

Matrix: Water

Compound	RL	Units	MDL	Units
PCB-1016	1	ug/L	0.17	ug/L
PCB-1221	1	ug/L	0.13	ug/L
PCB-1232	1	ug/L	0.16	ug/L
PCB-1242	1	ug/L	0.22	ug/L
PCB-1248	1	ug/L	0.1	ug/L
PCB-1254	1	ug/L	0.16	ug/L
PCB-1260	1	ug/L	0.17	ug/L

Matrix: Solid

Compound	RL	Units	MDL	Units
PCB-1016	33	ug/kg	21	ug/kg
PCB-1221	33	ug/kg	16	ug/kg
PCB-1232	33	ug/kg	14	ug/kg
PCB-1242	33	ug/kg	13	ug/kg
PCB-1248	33	ug/kg	17	ug/kg
PCB-1254	33	ug/kg	17	ug/kg
PCB-1260	33	ug/kg	17	ug/kg

APPENDIX E

Field Audit Checklist

APPENDIX E

FIELD AUDIT CHECKLIST

Briefing with Project Manager

SIGNATURE OF AUDITOR_____ DATE OF AUDIT _____

PROJECT MANAGER _____ PROJECT NO. _____

PROJECT LOCATION_____

TYPE OF INVESTIGATION _____
(authority, agency)

Yes ____ No ____ N/A ____ 1. Was a project plan prepared? If yes, what items are addressed in the Plan?

_____

_____

_____

Yes ____ No ____ N/A ____ 2. Were additional instructions given to project participants (i.e., changes in the project plan)? If yes, describe these changes.

_____

_____

_____

Yes ____ No ____ N/A ____ 3. Is there a written list of sampling locations and descriptions? If yes, describe where documents are.

_____

_____

_____

Yes ____ No ____ N/A ____ 4. Is there a map of sampling locations? If yes, where is the map.

_____

_____

_____

FIELD AUDIT CHECKLIST

Field Observations

Yes _____ No _____ N/A ____ 1. Are the number, frequency and types of field measurements and observations taken as specified in the project plan or as directed by the project manager? If yes, where are they recorded?

Yes _____ No _____ N/A ____ 2. Are samples collected in the types of containers specified for each type of analysis? If no, what kind of sample containers are used?

Yes _____ No _____ N/A ____ 3. Are samples preserved as required? If no or N/A, explain.

Yes _____ No _____ N/A ____ 4. Are the number, frequency and types of samples collected as specified in the project plan or as directed by the project manager? If no, explain why not.

Yes _____ No _____ N/A ____ 5. Are samples packed for preservation when required (i.e., packed in ice, etc.)? If no or N/A, explain why.

Yes _____ No _____ N/A ____ 6. Is sample custody maintained? How?

FIELD AUDIT CHECKLIST

Document Control

Yes _____ No _____ N/A ____ 1. Are samples identified with sample tags? If no, how are samples identified?

Yes _____ No _____ N/A ____ 2. Are sample tags completed (e.g., station no., location, date, time, analyses, signatures on samples, type, preservatives, etc.)? If yes, describe types of information recorded.

Yes _____ No _____ N/A ____ 3. Are samples collected listed on a chain-of-custody record? If yes, describe the type of chain-of-custody record used and what information is recorded.

Yes _____ No _____ N/A ____ 4. If used, are sample tag numbers recorded on chain-of-custody documents?

Yes _____ No _____ N/A ____ 5. Does information on sample tags and chain-of-custody records match?

Yes _____ No _____ N/A ____ 6. Does the chain-of-custody record indicate the method of sample shipment?

Yes _____ No _____ N/A ____ 7. Is the chain-of-custody record included with the samples in the shipping container?

Yes _____ No _____ N/A ____ 8. If used, do the sample traffic reports agree with the sample tags?

Yes _____ No _____ N/A ____ 9. If used, are blank samples identified?

Yes _____ No _____ N/A ____ 10. If collected, are duplicate samples identified on sample tags and chain-of-custody records?

Yes _____ No _____ N/A ____ 11. If used, are spiked samples identified?

Yes _____ No _____ N/A ____ 12. Are logbooks signed by the individual who checked out the logbook from the project coordinator?

Yes _____ No _____ N/A ____ 13. Are logbooks dated upon receipt from the project coordinator?

Yes _____ No _____ N/A ____ 14. Are logbooks project-specific (by logbook or by page)?

Yes _____ No _____ N/A ____ 15. Are logbook entries dated and identified by the author?

Yes _____ No _____ N/A ____ 16. Are photographs documented in logbooks (e.g., time, date, description of subject, photographer, etc.)?

Yes _____ No _____ N/A ____ 17. If film from a self-developing camera is used, are photos matched with logbook documentation?

Yes _____ No _____ N/A ____ 18. Are sample tag numbers recorded? If yes, describe where they are recorded?

Yes _____ No _____ N/A ____ 19. Are calibration of pH meters, conductivity meters, etc., documented? If yes, describe where this is documented.

Yes _____ No _____ N/A ____ 20. Are amendments to the project plan documented? If yes, describe where the amendments are documented.

FIELD AUDIT CHECKLIST

Debriefing with Project Manger

Yes _____ No _____ N/A ____ 1. Was a debriefing held with project manager and/or other participants?

Yes _____ No _____ N/A ____ 2. Were any recommendations made to the project participants during the debriefing? If yes, list recommendations.

APPENDIX F

Laboratory Audit Checklist

APPENDIX F

LABORATORY AUDIT CHECKLIST

The attached laboratory audit checklist is designed for the completion of an on-site laboratory audit primarily in support of an ongoing Haley & Aldrich, Inc (Haley & Aldrich). project or Master Agreement. The checklist consists of a number of charts that will be used to audit the laboratory against the project Quality Assurance Project Plan (QAPP) standard operating procedures (SOP) and/or standard U.S. EPA Methods. Only those checklists that pertain to the project or program of interest shall be covered during the on-site laboratory audit.

The laboratory audit checklist covers the 13 sections detailed below:

1. General Laboratory Information
2. Personnel
3. laboratory Environmental/Facilities
4. equipment and Instrumentation
5. Field Sampling
6. Sample Receipt and Storage
7. Organic Analyses by Gas Chromatography (GC)
8. Organic Analyses by GC/Mass Spectrometry
9. Metals Analysis
10. Other Classical Analyses
11. Data Reduction, Review and Reporting
12. Performance and System Audits

The attached charts (CHARTS I – BB) contain lists on which the availability of written operating procedures is to be checked. These lists cover instrument maintenance and calibration, all aspects of sampling, and the quality control program of the laboratory. The laboratory should be asked to show supporting documents to the auditor(s) during the on-site inspection and to discuss them with the auditor(s).

These checklists are not a demand or request for written procedures (for example, a Quality Control Program) in a particular standard format. The key point is that the principal laboratory controls should be documented in a permanent way. Some procedures may be brief or may not include all of the items to be checked.

GENERAL LABORATORY INFORMATION

1. Laboratory Name: _____

2. Street Address: _____

_____

Mailing Address: _____

_____

3. Telephone No.: _____ FAX No,: _____

4. Number of Employees: _____ Years in Operation: _____

5. Name of laboratory Director: _____

Name of Laboratory Project Manager: _____

Name of Quality Assurance (QA) Officer: _____

6. Project Details:

Project Number: _____ Project Name: _____

Laboratory analyses required for project:

Parameters	Matrices	Methods

Completed by: _____ _____ Date: _____

NAME TITLE

1.0. AUDIT CHECKLIST – GENERAL LABORATORY INFORMATION

CHART I

	ITEM	YES	NO	COMMENT
	Does the lab maintain a Quality Assurance Manual?			
	Does the manual address the important elements of a QC program, including the following:			
a.	Project Organization and Responsibility			
b.	Organization Chart?			
c.	Personnel Training/Certification?			
d.	Facilities and Equipment?			
e.	Quality Assurance Objectives?			
f.	Sampling Procedures?			
g.	Sample Custody?			
h.	Sample Receiving and Disposal?			
i.	Analytical Procedures?			
j.	Calibration Procedures and Frequency			
k.	Preventive Maintenance?			
l.	Certification of Reagents and Standards?			
m.	Internal Quality Control Checks?			
n.	Data Reduction, Review and Reporting?			
o.	Reliability of Data?			
p.	Feedback and Corrective Action?			
q.	Record keeping and Storage?			
r.	Performance and System Audits?			
s.	Quality Control Reports?			

1.0. AUDIT CHECKLIST – GENERAL LABORATORY INFORMATION

CHART I

ITEM	YES	NO	COMMENT
Is the manual available to all laboratory personnel?	____	___	_____
Is the manual updated regularly with appropriate document control procedures?	____	___	_____
Is line authority for all referenced responsibilities explained by inclusion in an organization chart(s)?	____	___	_____
Is the organizational structure appropriate to accomplish the project QA objectives?	____	___	_____
Are QA/QC responsibilities and reporting relationships clearly defined?	____	___	_____
Are all staff aware of QA/QC and its application?	____	___	_____
Is the QA Officer a full-time function?	____	___	_____
Does the QA Officer operate independently of the analyses?	____	___	_____
Does the QA Officer report directly to senior management?	____	___	_____
Are internal QA reviews conducted at least annually and recorded including any corrective action taken?	____	___	_____
QA Objectives and Criteria:			
a. Are the terms and definitions for precision, accuracy, comparability, representativeness and completeness properly used,	____	___	_____
b. Have the following been defined for each matrix and parameter:			
1) Level of QA effort (frequency of QC, etc.)?	____	___	_____
2) Accuracy (matrix spikes, surrogate spikes, reference samples, etc.)?	____	___	_____
3) Precision (replicate samples)?	____	___	_____

1.0. AUDIT CHECKLIST – GENERAL LABORATORY INFORMATION

CHART I

ITEM	YES	NO	COMMENT
4) Sensitivity or MDL?	_____	___	_____
5) Statistical reporting limits?	_____	___	_____
c. Are quantitation limits established for each analytical parameter?	_____	___	_____
d. Are field and lab analyses both covered?	_____	___	_____
e. If appropriate, are completeness objectives quantitatively stated?	_____	___	_____
f. Are representativeness and comparability appropriately addressed?	_____	___	_____
Do SOPs include or refer to SOPs addressing in following areas:			
a. Interferences?	_____	___	_____
b. Safety Considerations?	_____	___	_____
c, Apparatus and Equipment?	_____	___	_____
d. Reagents and Supplies?	_____	___	_____
e. Sample Preservation and Storage?	_____	___	_____
f. Sample Preparation?	_____	___	_____
g. Instrumentation Calibration?	_____	___	_____
h. Quality Control Procedures?	_____	___	_____
i. Detailed Step-by-Step Procedures?	_____	___	_____
j. Sample Calculations?	_____	___	_____
k. Method Performance Criteria? (accuracy and precision)	_____	___	_____
Are there SOPs for:			
a. Sample collection, preservation, reception storage and handling?	_____	___	_____
b. documenting all analytical procedures?	_____	___	_____
c. Operation/calibration of instrument?	_____	___	_____

1.0. AUDIT CHECKLIST – GENERAL LABORATORY INFORMATION

CHART I

ITEM		YES	NO	COMMENT
d.	Preventive maintenance?	____	___	_____
e.	Quality Control for each type of test?	____	___	_____
f.	Quality Control Charting?	____	___	_____
g.	Record keeping and storage?	____	___	_____
h.	Data reduction, review and reporting	____	___	_____
i.	Personnel training/certification?	____	___	_____
j.	Procurement and inventory procedures?	____	___	_____
k.	Glassware cleaning?	____	___	_____
l.	Waste disposal?	____	___	_____

Is at least the following minimum QC practiced in the lab?

a. For Inorganic/Wet Chemistry:

1)	Minimum 3 concentrations of standards plus blank, and one check standard in 10; the lab shall repeat all samples if check standard is outside ±10% (±20% for mercury).	____	___	_____
2)	One method blank in 20 or per batch	____	___	_____
3)	One matrix spike in 20 or per batch	____	___	_____
4)	One lab duplicate or matrix spike duplicate in 20 per batch	____	___	_____
5)	One laboratory control sample (LCS) (consists of a control matrix spiked with analytes representative of the target analytes) in 20 or per batch.	____	___	_____

b. For Organic Chemistry:

1)	Minimum 5 concentrations of standards plus blank and one check standard in 10; if any are outside control limits, repeat all samples.	____	___	_____
2)	One method blank in 20 or per batch.	____	___	_____

1.0. AUDIT CHECKLIST – GENERAL LABORATORY INFORMATION

CHART I

ITEM	YES	NO	COMMENT
3) One matrix spike in 20 or per batch.			
4) One lab duplicate or matrix spike duplicate in 20 or per batch.			
5) Surrogates for all samples.			
Is a QC batch defined as a single event (i.e. one day or less)?			
Are there any exceptions to the above minimum QC practiced in the lab?			
Has the lab established control limits for all of the above types of QC samples?			
Are control limits at least as tight as those stated in the methods?			
Are quality control data (e.g. standard curve, results of duplicates and spikes) accessible for all analytical results?			
Are method detection limits determined and documented?			
Are control charts maintained for each routine analysis?			
Do lab records indicate what corrective action has been taken when results fail to meet QC criteria?			
Is a program of initial and continuing calibration established for each method?			
Does QA Plan include calibration documentation requirements:			
a. Date(s) of calibration?			
b. Identification of standards used?			
c. Personnel performing calibration?			
d. Results of calibration (raw data and summary statistics)?			
e. Corrective actions taken?			

1.0. AUDIT CHECKLIST – GENERAL LABORATORY INFORMATION

CHART I

ITEM	YES	NO	COMMENT
Are the primary reference standards used for calibration only?	____	___	_____
Are all working standards versus primary standards verifies and documented?	____	___	_____
Are reference materials (lab standards) traceable?	____	___	_____
Are reagent grade or higher purity chemicals used to prepare standard?	____	___	_____
Are fresh analytical standards prepared at a frequency consistent with good QC?	____	___	_____
Are reference materials/reagents properly labeled with concentrations, dates of receipt, preparation/expiration and the identity of the person preparing the reagent?	____	___	_____
Are updated equipment operating instructions available?	____	___	_____
Are all analytical procedures written as SOPs available for review?	____	___	_____
Are SOPs provided and maintained in the laboratory or area of use?	____	___	_____
Are all procedural steps and options described in SOPs?	____	___	_____
Are procedures in place for making and controlling revisions to SOPs?	____	___	_____
Are the criteria of method selection included (e.g., in order to obtain a specific Data Quality Objective)?	____	___	_____
If method choice is governed by regulatory requirement (e.g., NPDES, SDWA, RCRA) have the appropriate methods been chosen?	____	___	_____
Are approved methods being used as specified?	____	___	_____
Are procedures documented for data handling reporting and record keeping?	____	___	_____

1.0. AUDIT CHECKLIST – GENERAL LABORATORY INFORMATION

CHART I

ITEM	YES	NO	COMMENT
Are documented validation procedures applied at appropriate levels of all measurement procedures?	____	___	_____
Are documented procedures available for checking the validity of reported analysis values?	____	___	_____
Are predetermined limits available for data acceptability beyond which corrective action is required?	____	___	_____
Are documented procedures available for correcting erroneously reported results?	____	___	_____
Does the lab have a LIMS currently in use? If yes, manufacturer: _____ Model No: _____	____	___	_____

Brief Description: _____

_____

_____

_____

_____

_____

Does the LIMS have an audit trail feature: ____ ___ _____

Completed by: _____ _____ Date: _____

NAME TITLE

2.0. AUDIT CHECKLIST – PERSONNEL

CHART J

ITEM	YES	NO	COMMENT
Are there documented protocol for training in quality control methods?	____	____	_____
Do personnel assigned to this project have the appropriate background to successfully accomplish the objectives of this project?	____	____	_____
Does each analyst accountable for performing tasks in any of the following areas meet the specified minimum experience:			
a. General chemistry and instrumentation – 6 months.	____	____	_____
b. Sample preparation – 6 months.	____	____	_____
c. Gas chromatography – 1 year.	____	____	_____
d. Mass spectrometry – 1 year.	____	____	_____
e. Atomic absorption – 1 year.	____	____	_____
f. Atomic emission – 1 year.	____	____	_____
g. spectral/chromatography interpretation – 2 years.	____	____	_____
h. pesticide residue analysis – 2 years.	____	____	_____
Is backup provided for technical staff?	____	____	_____
Are qualifications, training and experience recorded?	____	____	_____
Is there documented evidence of analyst proficiency for each test method performed?	____	____	_____
Is the QA Officer familiar with all test procedures and QC requirements?	____	____	_____
Does the lab have an in-house training program or send staff to training school?	____	____	_____

Completed by: _____ _____ Date: _____

NAME TITLE

3.0. AUDIT CHECKLIST – LABORATORY ENVIRONMENTAL/FACILITIES

CHART K

ITEM	YES	NO	COMMENT
Does the lab building have a security system?	____	____	_____
Is access to the laboratory area controlled?	____	____	_____
Is equipment protected and environment monitored as needed?	____	____	_____
Does the lab have adequate work space, ventilation, light and access to stable power sources at workstation?	____	____	_____
Is the lab free of dust, drifts and temperature extremes?	____	____	_____
Is water free of trace contamination available for preparation of standards and blanks?	____	____	_____
Is the pH and conductivity of water routinely checked and recorded on a daily basis?	____	____	_____
Are exhaust hoods provided to allow contamination-free work with volatile and hazardous materials?	____	____	_____
Is the air flow of the hoods periodically checked and recorded?	____	____	_____
Are adequate facilities, including cold storage, provided for separate storage of samples, extracts, reagents, solvents, reference materials and standards to preserve their identity, concentration, purity and stability?	____	____	_____
Is adequate chemical storage space available and are chemicals properly segregated according to hazard class?	____	____	_____
Are solvent storage cabinets properly vented as appropriate for the prevention of possible lab contamination?	____	____	_____
Does the lab have adequate safety devises such as eye wash stations, spill control stations, showers, first-aid stations, (etc.)?	____	____	_____

3.0. AUDIT CHECKLIST – LABORATORY ENVIRONMENTAL/FACILITIES

CHART K

ITEM	YES	NO	COMMENT
Are these safety devises checked routinely and documented to ensure they are still working properly?	____	____	_____
Are special facilities provided for handling extremely toxic materials such as dioxin (e.g., glove box, controlled air)?	____	____	_____
Are chemical waster disposal policies and procedures well-defined and followed by the lab?	____	____	_____

Completed by: _____ _____ Date: _____

NAME TITLE

4.0. AUDIT CHECKLIST – EQUIPMENT AND INSTRUMENTATION

CHART L

ITEM	YES	NO	COMMENT
Is appropriate equipment/instrumentation available for use in accordance with required methodology?	____	____	_____
Is equipment adequately maintained with sufficient spare parts and are maintenance instructions available?	____	____	_____
Is out-of-service equipment clearly labeled?	____	____	_____
Are instrument maintenance logs kept?	____	____	_____
Are standard curves prepared to adequately cover the expected concentration ranges of samples?	____	____	_____
Are calibration logs maintained?	____	____	_____
Is a new curve prepared annually (or more frequently as specified by the method) or whenever new reagents are prepared, whichever is more frequent?	____	____	_____
Are calibration labels used as applicable?	____	____	_____
Is proper backup equipment available?	____	____	_____
General Equipment Requirements:			
Analytical and Pan Balances:			
a. Are analytical balances capable of weighing 0.1 mg in use?	____	____	_____
b. Is a record of balance calibration kept for at least two ranges with Class S weights? (Please specify the ranges)	____	____	_____
c. Do records show daily functional and calibration checks for analytical balances and weekly for pan balances?	____	____	_____
d. Are the balances services and calibrated at least annually?	____	____	_____

4.0. AUDIT CHECKLIST – EQUIPMENT AND INSTRUMENTATION

CHART L

ITEM	YES	NO	COMMENT
pH Meter:			
a. Is a clean pH meter and appropriate electrode with scale gradations at least 0.1 pH units in use?	____	____	_____
b. Is a thermometer or temperature sensor for automatic compensation in use.	____	____	_____
c. Do records show daily, or before each use, calibration, whichever is less frequent?	____	____	_____
d. Are aliquots of standard pH 4 & pH 7, or pH 7 & pH 10 used only once?	____	____	_____
Conductivity Mater:			
a. Are a conductivity meter and probe of sufficient sensitivity in use?	____	____	_____
b. Do records show a daily, or before each use, calibration check, whichever is less frequent?	____	____	_____
c. Do records show cell constant is determined annually?	____	____	_____
Glassware:			
a. Is glassware cleaned in a manner appropriate for the analytical procedures for which it is used?	____	____	_____
b. Are glassware cleaning SOPs available where cleaning occurs?	____	____	_____
c. Is glassware maintained separately for each department?	____	____	_____
Refrigeration:			
a. Is a thermometer in each refrigerator with a bulb immersed in liquid?	____	____	_____
b. Are thermometers graduated in increments no lager than $1^{o}C$?	____	____	_____

4.0. AUDIT CHECKLIST – EQUIPMENT AND INSTRUMENTATION

CHART L

ITEM	YES	NO	COMMENT
c. Are thermometers calibrated against NIST (NBS) – traceable thermometer and documented?	____	____	_____
d. Do records show that refrigerator temperatures are maintained in the range of 2^{o} to 6^{o}C?	____	____	_____
e. Are temperatures for each refrigerator recorded daily?	____	____	_____
f. Are samples stored in separate refrigerators from standards?	____	____	_____
Ovens:			
a. Are thermometers graduated in increments no larger than 1^{o}C?	____	____	_____
b. Are thermometers calibrated against NIST (NBS) – traceable thermometer and documented?	____	____	_____
c. If the oven temperature cannot be read without opening the door, is the bulb of the thermometer in a sand bath?	____	____	_____
d. Is oven temperature adequately monitored (e.g., beginning and end of each use cycle)?	____	____	_____
Thermometers:			
a. Does the lab have access to a NIST (NBS) traceable thermometer?	____	____	_____
b. Is the calibration of each working mercury-in-glass thermometer checked at least annually against a NIST (NBS) traceable certified thermometer?	____	____	_____
c. Is the calibration of each dial0type thermometer checked at least quarterly against a NIST (NBS)-traceable thermometer?	____	____	_____

Completed by: _____ _____ Date: _____

NAME TITLE

5.0. AUDIT CHECKLIST – FIELD SAMPLING: (Complete if this lab will conduct field sampling for Haley & Aldrich).

CHART M

ITEM	YES	NO	COMMENT
Is the Site Specific Quality Assurance Project Plan (QAPP) available to field personnel	____	____	_____
Are field personnel familiar with the QC requirements of the Site specific QAPP	____	____	_____
Do field documentation procedures:			
a. document source of reagents/supplies?	____	____	_____
b. Include procedures/forms for recording the exact location and specific considerations associated with sample collection?	____	____	_____
c. Document specific preservative methods?	____	____	_____
d. Include labels containing all necessary information?	____	____	_____
e. Include forms for tracking sample custody?	____	____	_____
Do sampling procedures follow contract specifications?	____	____	_____
Is there a unique identification number on each sample?	____	____	_____
Is sampling information properly recorded such as sample ID, (grab vs. composite), preservatives, analytes, location, date and time of collection and name of supplier?	____	____	_____
Are written chain-of-custody procedures available for review? Are they in accordance with EPA and QAPP guidelines?	____	____	_____
Are there written sampling procedures covering sampling plans, sampling equipment, sample collections, preservation, identification, storage and lab handling?	____	____	_____

5.0. AUDIT CHECKLIST – FIELD SAMPLING: (Complete if this lab will conduct field sampling for Haley & Aldrich).

CHART M

ITEM	YES	NO	COMMENT
Are there written descriptions of the chain-of-custody of samples? (Attach a copy of chain-of-custody form).	____	____	_____
Are there written procedures for field measurement of flow, dissolved oxygen, residual chlorine, etc.?	____	____	_____
Are there written SOPs for monitoring water supply, effluent, ambient air, stacks, mobile vehicles, pesticides, radiation, etc.?	____	____	_____
Are proper preservation techniques being used for the analytical methods and sample types concerned?	____	____	_____
Are provisions made for the collection of QA/QC split samples?	____	____	_____
Are provisions made for field blanks and duplicate samples at an appropriate percentage (normally 10% or minimum of one per matrix type, whichever is greater, or as specified in Contract)?	____	____	_____
Are adequate facilities available to do compatibility testing?	____	____	_____

Completed by: _____ _____ Date: _____

NAME TITLE

6.0. AUDIT CHECKLIST – SAMPLE RECEIPT/STORAGE

CHART N

ITEM	YES	NO	COMMENT
Are appropriate chain-of-custody procedures documented and followed in the lab?	____	____	_____
Are there adequate written procedures for receipt and storage of samples to ensure that holding times are met?	____	____	_____
Do lab custody procedures:			
a. Identify sample custodian? Name of custodian: _____	____	____	_____
b. Provide for custody record within the lab?	____	____	_____
c. Specify procedures for sample handling, storage, dispersement for analysis, and disposal?	____	____	_____
Does a permanent record exist for sample log-in?	____	____	_____
Are samples assigned an unambiguous sample number when logged in?	____	____	_____
Are there sample preservation records?	____	____	_____
Are the pH values of aqueous samples checked and adjusted in a hood for trace metals, phenols, cyanide and TRPH during sample log-in?	____	____	_____
Is the temperature of sample cooler/shuttle recorded upon receipt?	____	____	_____
Is client notified of receipt temperature outside of $4^{o} \pm 2^{o}C$?	____	____	_____
Are discrepancies between sample labels and chain-of-custody documents recorded?	____	____	_____
Is client notified of any discrepancies following sample receipt and log-in?	____	____	_____
Are there adequate facilities for sample receipt and storage of samples?	____	____	_____

6.0. AUDIT CHECKLIST – SAMPLE RECEIPT/STORAGE

CHART N

ITEM	YES	NO	COMMENT
Are samples stored in such a way as to maintain their identity, integrity, stability and concentration?	____	____	_____
How long will the lab store samples before disposing?	____	____	_____

Completed by: _____ _____ Date: _____

NAME TITLE

7.0. AUDIT CHECKLIST – ORGANIC ANALYSES BY GAS CHROMATOGRAPHY (GC):

CHART O

A. Summary of GC Staff:

Position Title	Name of Employee	Years of Experience [1]	Education in Chemistry	Appropriate Training in Analytical Testing Area [2]
GC Group Leader [3] (experience: 2 years minimum required)				
GC Operators [3] (Experience: 1 year minimum required)				
GC Interpretation Experts [3] (Experience: 2 years minimum required)				
PCB and Pesticide Residue Analysis Experts [3] (Experience: 2 years minimum required)				
Purge & Trap Experts [3] (Experience: 1 year minimum required)				
Extraction & Concentration Experts [4] (Experience: 6 months minimum required)				

1. Requirements for experience as listed are minimal and maybe increased for specific projects involving difficult samples or unusual matrices.
2. Manufacturer sponsored class, EPA symposiums, etc.
3. Minimum of Bachelor's degree in chemistry or any scientific/engineering discipline.
4. Minimum of high school diploma and knowledge of general chemistry.

7.0 AUDIT CHECKLIST – ORGANIC ANALYSES BY GC:

CHART P

B. Summary of GC Instrumentation:

No.	Manufacturer	Model Number	Year Acquired	Used for Method [1]	Comments [2]	Haley & Aldrich Use Only S	U
1.							
2.							
3.							
4.							
5.							
6.							
7.							
8.							
9.							
10.							
11.							
12.							
13.							
14.							
15.							
16.							
17.							
18.							
19.							
20.							

1. State EPA method from SW-846 8000 or 40 CFR 136 600 series
2. Detectors, condition, autosamplers, modification, etc.

7.0 AUDIT CHECKLIST – ORGANIC ANALYSES BY GC:

CHART Q

ITEM	YES	NO	COMMENT
C. Questions about the GC instruments described above. (If one answer does not apply to all of the instruments listed, use the instrument number (1-20) to designate answers as appropriate.			
Are the following detectors available and are they used appropriately?			
a. Flame ionization (603, 604, 609, 8015, 8030, 8040, 8060, 8090, 8100)?	____	____	_____
b. Photoionization (602, 8020)?	____	____	_____
c. Electron capture (604, 606, 608, 609, 612, 8040, 8060, 8080, 8090, 8120, 8150)?	____	____	_____
d. Electrolytic conductivity (601, 611, 8010, 8080, 8140)?	____	____	_____
e. Microcoulometric (601, 611, 8140)?	____	____	_____
f. Thermal Energy Analyzer (607)?	____	____	_____
g. Nitrogen/Phosphorus (607, 8140)?	____	____	_____
h. Flame photometric (8140)?	____	____	_____
Are the column ovens, at a minimum, capable of control within $+0.2^{o}$ C to 220^{o} C?	____	____	_____
Are the injection ports glass lined?	____	____	_____
Are the purge and trap systems subjected to a periodic bake-out and cleaning process and are these actions documented?	____	____	_____
Will the system accept 5mL samples?	____	____	_____
Is the gaseous headspace less than 15mL?	____	____	_____
Is the trap a minimum of 25 cm long?	____	____	_____
Will the system accept 25mL samples?	____	____	_____
Are appropriate confirmation columns or GC/MS analysis in use?	____	____	_____
Is methanol purge and trap quality or equivalent?	____	____	_____
Id calibration by internal standards?	____	____	_____

7.0 AUDIT CHECKLIST – ORGANIC ANALYSES BY GC:

CHART Q

ITEM	YES	NO	COMMENT
If not, is calibration by external standards?	____	____	_____
Is the calibration curve or calibration factor verified every 10 samples?	____	____	_____
Is there a calibration protocol available to the operator?	____	____	_____
Are calibration results kept in a permanent logbook?	____	____	_____
Are SOPs available for each analytical method?	____	____	_____
Are 5-point calibrations used?	____	____	_____
Are surrogate recoveries determined for each sample?	____	____	_____
Is corrective action taken if surrogate recoveries exceed QC limits?	____	____	_____
Does laboratory use EPA CLP protocols to evaluate internal QC data?	____	____	_____
If not, what are the laboratory's criteria for evaluation of internal QC data?	____	____	_____
Are EPA check samples analyzed as appropriate?	____	____	_____
Is a matrix spike and a matrix spike duplicate run with each batch?	____	____	_____
Is corrective action taken if matrix spike recoveries exceed QC limits?	____	____	_____
Are method blanks run with each batch?	____	____	_____
Has method proficiency (acceptable accuracy and precision) been established for all procedures?	____	____	_____
Are method matrix-specific accuracy criteria established and maintained?	____	____	_____
Have laboratory-specific surrogate percent recovery QC limits been established?	____	____	_____
Are manufacturer's operating manuals readily available to the operator?	____	____	_____

7.0 AUDIT CHECKLIST – ORGANIC ANALYSES BY GC:

CHART Q

ITEM	YES	NO	COMMENT
Is a permanent logbook kept for each instrument that summarizes instrument problems and servicing records?	____	____	_____
Have any instruments been modified in any way?	____	____	_____
Are the instruments properly vented?	____	____	_____

Completed by: _____ _____ Date: _____

NAME TITLE

8.0 AUDIT CHECKLIST – ORGANIC ANALYSES BY GC/MASS SPECTROMERY

CHART R

A. Summary of GC/MS Staff:

Position Title	Name of Employee	Years of Experience [1]	Education in Chemistry	Appropriate Training in Analytical Testing Area [2]
GC/MS Group Leader [3] (Experience: 2 years minimum required)				
GC/MS Operator [3] (Experience: 1 year minimum required)				
GC/MS Spectral Interpretation Experts [3] (Experience: 2 years minimum required				
Purge & Trap Experts [3] (Experience: 1 year minimum required)				
Extraction and Concentration Experts [4] (Experience: 6 months minimum required)				

1. Requirements for experience as listed are minimal and maybe increased for specific projects involving difficult samples or unusual matrices.
2. Manufacturer sponsored class, EPA symposiums, etc.
3. Minimum of Bachelor's degree in chemistry or any scientific/engineering discipline.
4. Minimum of high school diploma and knowledge of general chemistry.

8.0 AUDIT CHECKLIST – ORGANIC ANALYSES BY GC/MASS SPECTROMERY

CHART S

B. Summary of GC/MS Instrumentation:

No.	Manufacturer	Model Number	Year Acquired	Used for Method [1]	Comments [2]	Haley & Aldrich Use Only S	U
1.							
2.							
3.							
4.							
5.							
6.							
7.							
8.							
9.							
10.							
11.							
12.							
13.							
14.							
15.							
16.							
17.							
18.							
19.							
20.							

1. State EPA method from SW-846 8000 or 40 CFR 136 600/1600 series
2. Condition, autosamplers, modification, etc.

8.0 AUDIT CHECKLIST – ORGANIC ANALYSES BY GC/MASS SPECTROMERY

CHART T

ITEM	YES	NO	COMMENT
C. Questions about the GC/MS instruments described above. (If one answer does not apply to all of the instruments listed, use the instrument number (1-20) to designate answers as appropriate.			
Are any GCs not programmable? If so, list the instrument number?	____	____	_____
Is a split/splitless capillary injection in place?	____	____	_____
Is the MS capable of scanning from 35 to 500 amu every one second or less?	____	____	_____
Is tuning compound, PFTBA, used to verify mass calibration?	____	____	_____
Do the mass spectra of BFP and DFTPP meet all criteria before each batch is run?	____	____	_____
Are standards containing all of the analytes of interest analyzed to verify response factors and update retention time?	____	____	_____
Is glassware heated at 400^{o}C in a muffle furnace after careful cleaning?	____	____	_____
Is glassware sealed and stored in a clean environment?	____	____	_____
Are magnetic media stored in a secure area?	____	____	_____
Are extensive in-house replacements parts available?	____	____	_____
Are manufacturer's operating manuals readily available to the operator?	____	____	_____
Is there a calibration protocol available to the operator?	____	____	_____
Are calibration results kept in a permanent logbook?	____	____	_____
Are SOPs available for each analytical method?	____	____	_____
Is a permanent logbook kept for each instrument that summarizes instrument problems and servicing records?	____	____	_____
Has the instrument been modified in any way?	____	____	_____
Are the instruments properly vented?	____	____	_____

8.0 AUDIT CHECKLIST – ORGANIC ANALYSES BY GC/MASS SPECTROMERY

CHART T

ITEM	YES	NO	COMMENT
Are 5-point calibrations used?	____	____	_____
Are continuing calibration checks done on a 12-hour basis?	____	____	_____
Are system performance response factors checked on a 12 hour basis?	____	____	_____
Are BFB and DFTPP tuning checks done on a 12-hour basis?	____	____	_____
Are DDT, pentachlorophenol and benzidine included in the DFTPP tuning solution?	____	____	_____
Are low-level methods routinely used for environmental soil/sediment samples?	____	____	_____
Is the most recent version of the EPA/NIST Mass Spectral Library available?	____	____	_____
For tentatively identified compounds, are library searches done for the 10 volatile organics and the 20 semivolatile organics of highest concentration?	____	____	_____
Are surrogate recoveries determined for each sample?	____	____	_____
Is a corrective action taken if surrogate recoveries exceed QC limits?	____	____	_____
Is a matrix spike and a matrix spike duplicate run with each batch?	____	____	_____
Is a corrective action taken if matrix spike recoveries exceed QC limits?	____	____	_____
Is a method blank run with each batch?	____	____	_____
Are samples screened by GC prior to GC/MS analysis?	____	____	_____
Has the laboratory demonstrated initial method proficiency?	____	____	_____
Are method matrix-specific accuracy criteria established and maintained?	____	____	_____

8.0 AUDIT CHECKLIST – ORGANIC ANALYSES BY GC/MASS SPECTROMERY

CHART T

ITEM	YES	NO	COMMENT
Have laboratory-specific surrogate percent recovery QC limits been established?	____	___	_____

Completed by: _____ _____ Date: _____

NAME TITLE

9.0 AUDIT CHECKLIST –METALS ANALYSIS:

CHART U

A. Summary of AA/ICP Staff:

Position Title	Name of Employee	Years of Experience [1]	Education in Chemistry	Appropriate Training in Analytical Testing Area [2]
Metals Group Leader [3] (Experience: 2years minimum required)				
ICP Spectroscopists [3] (Experience: 1 year minimum required)				
Flameless AA Spectroscopists [3] (Experience: 1 year minimum required)				
Flame and Cold Vapor AA Spectroscopists [4] (Experience: 1 year minimum require)				
Inorganic Sample Preparation Experts [4] (Experience: 6 months minimum required)				

9.0 AUDIT CHECKLIST –METALS ANALYSIS:

CHART U

Position Title	Name of Employee	Years of Experience [1]	Education in Chemistry	Appropriate Training in Analytical Testing Area [2]
Wet Lab Manager [3] (Experience: 2 years minimum required)				
UV/VIS Spectroscopist [3] (Experience: 6 months minimum required)				

1. Requirements for experience as listed are minimal and maybe increased for specific projects involving difficult samples or unusual matrices.
2. Manufacturer sponsored class, EPA symposiums, etc.
3. Minimum of Bachelor's degree in chemistry or any scientific/engineering discipline.
4. Minimum of high school diploma and knowledge of general chemistry.

9.0 AUDIT CHECKLIST –METALS ANALYSIS:

CHART V

B. Summary of AA/ICP Instrumentation

No.	Manufacturer	Model Number	Year Acquired	Used for Method [1]	Comments [2]	Haley & Aldrich Use Only S	U
1.							
2.							
3.							
4.							
5.							
6.							
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8.							
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10.							
11.							
12.							
13.							
14.							
15.							
16.							
17.							
18.							
19.							
20.							

1. State EPA method SW-846 6000/7000 or 40 CFR 136 200 series
2. Condition, autosamplers, modification, etc.

9.0 AUDIT CHECKLIST –METALS ANALYSIS:

CHART W

ITEM	YES	NO	COMMENT
C. Questions about the AA/ICP instruments described above. (If one answer does not apply to all of the instruments listed, use the instrument number (1-20) to designate answers as appropriate.			
Atomic absorption spectrophotometer:			
a. Are fuels and oxidants commercial grade?	____	___	_____
b. Is there a filter moisture trap between the air source and the spectrometer	____	___	_____
c. Is nitrous oxide reagent grade?	____	___	_____
d. Are flash-back arrestors and heaters in use when needed?	____	___	_____
e. Are all lamps dated when received and first put into use?	____	___	_____
f. Are lamps available for all elements analyzed?	____	___	_____
g. Does the AA have a deuterium background correction system?	____	___	_____
h. Does the AA have a Zeeman background correction system?	____	___	_____
i. Does the AA have a Smith-Hieftje background correction system?	____	___	_____
ICP-Atomic emission spectrometer:			
a. Is background correction technique in use and documented according to sample matrix at least quarterly?	____	___	_____
b. Has the instrument detection limit for each element been established and documented at least annually?	____	___	_____
c. Where required, has the effect of high dissolved solids and/or acid concentration been controlled?	____	___	_____
d. Has salt build-up on the nebulizer been controlled?	____	___	_____
e. When a new matrix is encountered, is a serial dilution, spike addition, or an alternate method technique in use to eliminate potential interference?	____	___	_____

9.0 AUDIT CHECKLIST –METALS ANALYSIS:

CHART W

ITEM	YES	NO	COMMENT
f. Is the spectrometer equipped with an argon gas supply?	____	___	_____
g. Are ultra high purity grade nitric acid, hydrochloric acid and deionized or distilled water used for sample processing and preparation?	____	___	_____
Are manufacturer's operating manuals readily available to the operator?	____	___	_____
Is there a calibration protocol available to the operator?	____	___	_____
Are calibration results kept in permanent logbooks?	____	___	_____
Are SOPs available for each analytical method?	____	___	_____
Is a permanent logbook kept for each instrument that summarizes instrument problems and servicing records?	____	___	_____
Is ICP calibration checked using a blank and three standards prior to sample analysis? _____	____	_____	
Is ICP calibration verified every 10 samples and at the end of the analytical run, using a calibration blank and a check standard?	____	___	_____
Does the result of check standard agree within ±10% of expected value?	____	___	_____
Are interelement and background correction factors checked at the beginning and end of an analytical run or twice during every 8-hour work shift, whichever is more frequent? _____	____	_____	
Does the result of interelement check sample agree with ±20% of expected value?	____	___	_____
Are AA calibrated with a blank and three standards (five standards for mercury)?	____	___	_____
Is the AA calibration verified with a reference standard to within ±10% of expected value prior to sample analysis?	____	___	_____
Is the AA calibration verified every tenth sample with a mid-range of reference standard?	____	___	_____

9.0 AUDIT CHECKLIST –METALS ANALYSIS:

CHART W

ITEM	YES	NO	COMMENT
Does the results of these check standards agree within ±20% of expected value?	____	___	_____
Is a matrix spike duplicate or internal QC duplicate run with each batch?	____	___	_____
Is corrective action taken if matrix spike recoveries exceed QC limits?	____	___	_____
Is a corrective action taken if internal QC duplicate or MS/MSD RPDs exceed QC limits?	____	___	_____
Is an analytical (post-digestion) spike analyzed after each sample?	____	___	_____
Is the method of standard addition in use where needed?	____	___	_____
Has the instrument been modified in any way?	____	___	_____
Are the instruments properly vented?	____	___	_____

Completed by: _____ _____ Date: _____

NAME TITLE

10.0 AUDIT CHECKLIST –OTHER CLASSICAL ANALYSIS:

CHART X

ITEM	YES	NO	COMMENT

A. Summary of Classical Analysis Staff:

Position Title	Name of Employee	Years of Experience [1]	Education in Chemistry	Appropriate Training in Analytical Testing Area [2]
Cyanide Analyst [4] (Experience: 6 months minimum required)				
IR Spectroscopists [3] (Experience: 6 months minimum required)				
TPH Analyst [3] (418.1) (Experience: 6 months minimum required)				
HPLC Analysis Experts [3] (Experience: 1 year minimum required)				
Explosives Analyst [3]				
Ion Chromatographic Analysis Experts (Experience: 1 year minimum required)				
Common Ions Analyst [3] Radionuclides Analyst (Experience: 2 years minimum required)				

10.0 AUDIT CHECKLIST –OTHER CLASSICAL ANALYSIS:

CHART X

ITEM	YES	NO	COMMENT

Position Title	Name of Employee	Years of Experience [1]	Education in Chemistry	Appropriate Training in Analytical Testing Area [2]
Microbiological Analysis Experts (Experience: 1 year minimum required)				

Other Classical lab Analysis Experts: (experience: 6 months minimum required). Please specify position, title and analysis.

Position Title	Name of Employee	Years of Experience [1]	Education in Chemistry	Appropriate Training in Analytical Testing Area [2]

1. Requirements for experience as listed are minimal and maybe increased for specific projects involving difficult samples or unusual matrices.
2. Manufacturer sponsored class, EPA symposiums, etc.
3. Minimum of Bachelor's degree in chemistry or any scientific/engineering discipline.
4. Minimum of high school diploma and knowledge of general chemistry.

10.0 AUDIT CHECKLIST –OTHER CLASSICAL ANALYSIS:

CHART Y

B. Summary of Instrumentation

No.	Instrument/ Manufacturer	Model Number	Year Acquired	Used for Method [1]	Comments [2]	Haley & Aldrich Use Only S	U
1.							
2.							
3.							
4.							
5.							
6.							
7.							
8.							
9.							
10.							
11.							
12.							
13.							

1. General classical wet lab/water quality analysis methods.
2. Condition, automation, modifications, etc.

10.0 AUDIT CHECKLIST –OTHER CLASSICAL ANALYSIS:

CHART Z

C. Questions about instruments described above. (If one answer does not apply to all of the instruments listed, use the instrument number (1-13) to designate answers as appropriate.

ITEM	YES	NO	COMMENT
Is the wavelength accuracy and repeatability of all spectrophotometers checked at several wavelengths for each batch of samples?	____	____	_____
Is photometric accuracy and repeatability checked and documented with NIST (NBS) traceable standards?	____	____	_____
Is acid washed glassware retained for phosphorus analyses only?	____	____	_____
Is ammonia free water used in preparation of standards and samples for nitrogen analyses?	____	____	_____
Are manufacturer's operating manuals readily available to operator of each instrument?	____	____	_____
Is there a calibration protocol available to the operator?	____	____	_____
Are calibration results kept in permanent logbooks?	____	____	_____
Are SOPs available for each analytical method?	____	____	_____
Is a permanent logbook kept for each instrument that summarizes instrument problems and servicing records?	____	____	_____
Has any instrument been modified in any way?	____	____	_____
Are 4-point calibrations used?	____	____	_____
Are continuing calibration checks done on a regular basis?	____	____	_____
Are internal QC run per method requirements? _____	____	____	_____
Is a method blank used with each blank?	____	____	_____

Completed by: _____ _____ Date: _____

NAME TITLE

11.0 AUDIT CHECKLIST –DATA REDUCTION, REVIEW AND REPORTING

CHARTAA

ITEM	YES	NO	COMMENT
Are there documented procedures for internal field and lab checks of:			
a. Precision/accuracy?	____	____	_____
b. Routine duplicates, spikes and standard samples?	____	____	_____
c. Statistical methods, including control chart and computer methods?	____	____	_____
Is there a written description of the lab record system including data handling/calculation review and audit?	____	____	_____
Is there a written description of the lab reporting system?	____	____	_____
Is there a system in place that provides for retrievability and traceability of the sample source, analytical methods, results, person performing analysis and date?	____	____	_____
Are records and reports adequately secured for the required amount of time to ensure their integrity per regulatory or H&A contract requirements?	____	____	_____
Are all lab notebooks when filled, raw data, QC data, analytical reports and pertinent documents filed in a secure, controlled archives area?	____	____	_____
Are permanently bound notebooks with consecutively numbered pages being used?	____	____	_____
Is a unique serial number clearly displayed on each notebook cover or spine?	____	____	_____
Have entries been made in a permanent fashion and corrections made without obliterating original entries?	____	____	_____
Are notebooks appropriately controlled?	____	____	_____
Has the supervisor of each individual maintaining a notebook personally examined and reviewed each notebook periodically, signed and dated the review?	____	____	_____
Do the lab's reports accurately, clearly and			

11.0 AUDIT CHECKLIST –DATA REDUCTION, REVIEW AND REPORTING

ITEM	YES	NO	COMMENT
unambiguously present results and all other relevant information?	____	____	_____
Does each test report include the following information?			
a. name and address of laboratory	____	____	_____
b. Unique identification and page number?	____	____	_____
c. Name and address of client?	____	____	_____
d. Case narrative?	____	____	_____
e. Sample identification and description?	_____	____	_____
f. Dates of sample receipt and test performed, as appropriate?	____	____	_____
g. Identification of sample preparation and analysis methods used?	____	____	_____
h. Description of any deviations from test method?	____	____	_____
i. Disclosure of any subcontractor used?	_____	____	_____
j. Results including all method required QC data?	____	____	_____
k. Description of any problems or failures identified?	____	____	_____
Measurement uncertainty, if relevant?	____	____	_____

Completed by: _____ _____ Date: _____

NAME TITLE

12.0 AUDIT CHECKLIST –PERFORMANCE AND SYSTEM AUDITS:

CHART BB

ITEM	YES	NO	COMMENT
Are there audits for both field and lab activities?	____	____	_____
Are there designated persons who will conduct the audits?	____	____	_____
Is there a documented protocol which will be used for audits?	____	____	_____
Are acceptance criteria defined?	____	____	_____
Are audit reports prepared and distributed? To whom?	____	____	_____
Are the type and frequency of audit reports specified?	____	____	_____
Do the reports address:			
a. Status of project (time table)?	____	____	_____
b. Results of performance and system audits?	____	____	_____
c. Data quality assessment?	____	____	_____
d. Significant QA problems and proposed corrective action?	____	____	_____

Completed by: _____ _____ Date: _____
NAME TITLE

APPENDIX C

Health and Safety Plan

HALEY & ALDRICH, INC.
SITE-SPECIFIC HEALTH & SAFETY PLAN

For

Former Leeds Assembly Plant – Northern Parcel

South of 6817 Stadium Drive

Project/File No. 37294

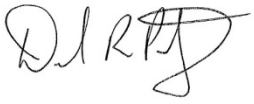
Prepared by: Dennis Kreitzburg Date: 11-17-2010

Revised by: Enter Revisor's Name Date: Enter Date

APPROVALS: The following signatures constitute approval of this Health & Safety Plan



Ana T. Mora - Regional H&S Coordinator — Date: 12/02/10



Daniel R. Putz - Site Project Manager — Date: 12/16/10

Scott A. Boston - Corporate H&S Manager — Date:
(Only required per request of RHSCs)

Date printed: 12/16/2010 at 5:11 PM

Note: This HASP has been developed for Haley & Aldrich purposes only and is not for use by others.

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APPENDIX A - HASP AMENDMENT FORM

APPENDIX B – ISSUANCE AND COMPLIANCE, SITE SAFETY OFFICER ROLES AND RESPONSIBILITIES, AND TRAINING REQUIREMENTS

Note: This HASP has been developed for Haley & Aldrich purposes only and is not for use by others.

1. PROJECT INFORMATION AND EMERGENCY RESOURCES

Project Name: Former Leeds Assembly-Northern Parcel	**H&A File No.: 37294**
Location: Enter Project Location	
Client/Site Contact: Phone Number: Emergency Phone Number:	David M. Favero, P.G. 217-522-6714 217-741-6235
General Contractor: **Superintendent:** Phone Number: Emergency Phone Number:	Enter General Contractor Name Enter Superintendent Name Enter Superintendent Phone Number Enter Superintendent Emergency Number
H&A Project Manager: Phone Number: Emergency Phone Number:	Daniel R. Putz 216-706-1314 216-410-4750
Regional Health & Safety Coordinator: Phone Number: Emergency Phone Number:	Ana T. Mora 913-217-6903 816-206-0791
Nearest Hospital: Address: (see map on next page) Phone Number:	Truman Medical Center Hospital Hill 2301 Holmes Street Kansas City, MO 64108 (816) 404-1000
Emergency Response Number:	**911**
Other Local Emergency Response Number:	Kansas City Police East Patrol 816-234-5530
Other Ambulance, Fire, Police, or Environmental Emergency Resources:	Inter City Fire Protection District 816-461-9090

Work Scope:

This Site-Specific Health and Safety Plan addresses the health and safety practices and procedures that will be employed by all Haley & Aldrich employees participating in the site characterization of the Project Site. This plan is based on an assessment of the site-specific health and safety risks available to Haley & Aldrich and Haley & Aldrich's experience with other project sites. The scope of work for the Site Characterization includes:

The soil investigation will be completed by installing 22 test pits. The total depth of the test pits will be approximately 1 to 3 feet into native soil unless the capability of the excavotor is exceeded by depth or obstructions. One to three soil samples will be collected from each test pit to be determined by field conditions such as: total depth, visual observations, and photoionization detector readings. The spoils from the test pits will be collected on polyethelene sheeting and the open excavation and spoils will be photographed. The spoils will be returned to the excavation, compacted by the excavator, and the pavement will be replaced. The soil samples will be sent to the analytical laboratory for analysis of VOCs, SVOCs, and the RCRA 8 Metals.

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The groundwater investigation will be completed by installing 22 shallow (up to 20 feet deep) and 2 deep wells (up to 35 feet deep). The monitoring wells will be installed in two rounds. The first round of wells will be installed, developed and sampled before installing the second round of wells to confirm if the locations are appropriate. The monitoring wells will be developed and sampled using low-flow methods for VOCs, SVOCs, and the RCRA 8 Metals.

Subcontractor(s) to be involved in on-site activities:

Firm Name	Work Activity
Enter Subcontractor Name	Enter Work Activity
Enter Subcontractor Name	Enter Work Activity

Projected Start Date: Enter Start Date

Projected Completion Date: Enter Completion Date

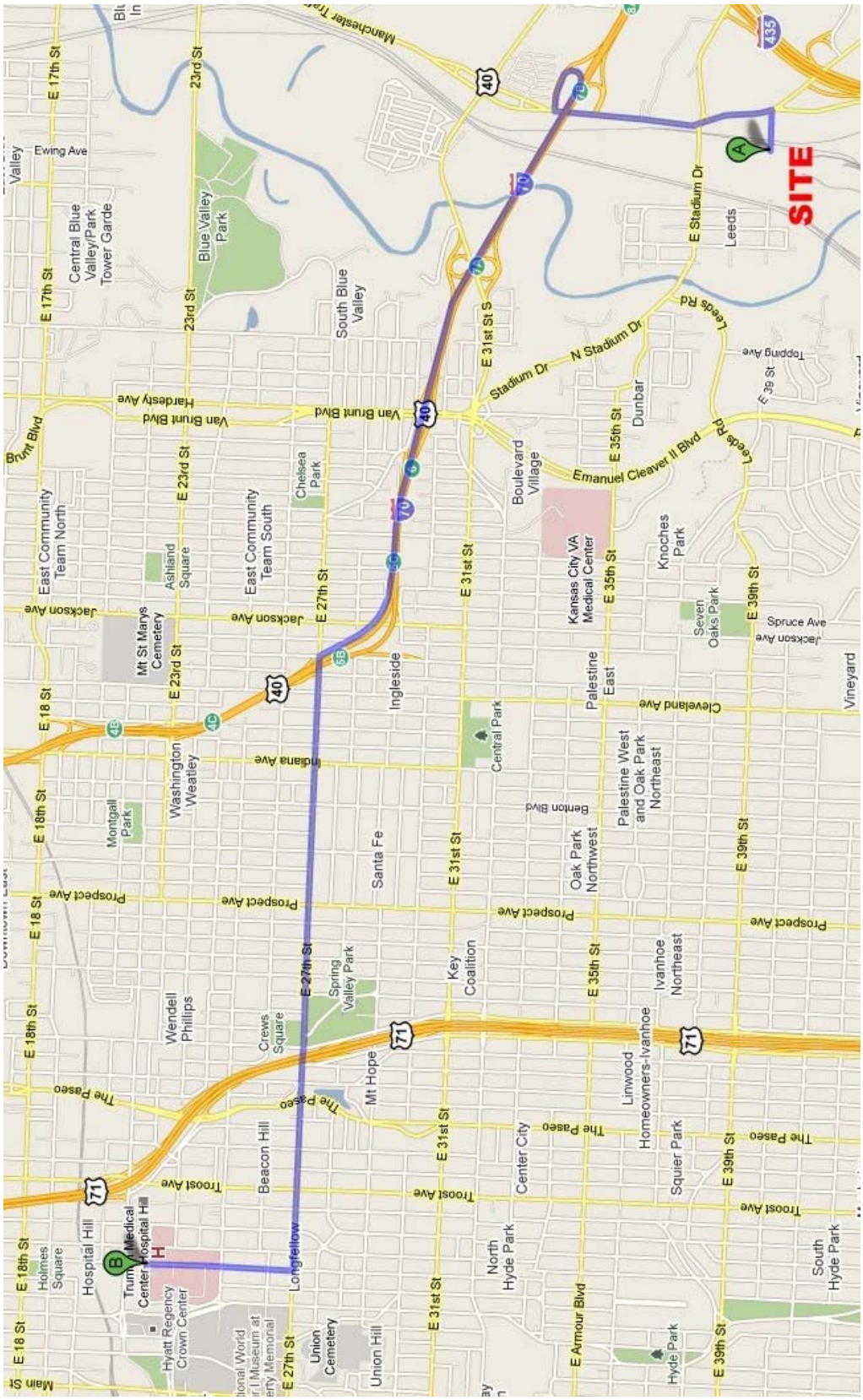
Estimated Number of Days to Complete Field Work: Enter Number of Days

Note: This HASP is developed for Haley & Aldrich purposes only and not for use by others.

Directions to the Nearest Hospital:

- Leave the site via the east entrance, you are on E 39th Street.
- Head east on E 39th Street until it ends and turn left (north) onto Raytown Road.
- Raytown Road becomes the Manchester Trafficway.
- After 0.7 miles turn right onto I-70 West.
- After 1.7 miles take the Jackson Avenue Exit (Exit 5C).
- At the end of the ramp merge onto E 29th Street heading west.
- E 29 Street becomes Myrtle Avenue.
- After 0.25 miles turn left (west) onto E 27th Street.
- After 2 miles turn right at Holmes Street.
- After 0.5 miles your have reached the Truman Medical Center at 2301 Holmes St, Kansas City, MO.

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Note: This HASP is developed for Haley & Aldrich purposes only and not for use by others.

2. SITE DESCRIPTION

Site Classification:

☑ Industrial	☐ Commercial	☐ Other Specify

General Description:

The approximate 15-acre Site is nearly entirely paved and is used for parking and storage. The one building on-site is a railroad siding shed. The site is in an industrial area.

Background and Historic Site Usage:

The Site was used for farming until the Leeds Assembly plant was constructed on the parcel adjacent to the north of the Site in 1928. Around that time railroads were constructed across the Site. The railroad siding shed near the eastern edge of the property boundary was built between 1957 and 1965. The former propand storage area in the northwest corner of the site was built between 1969 and 1978. Fill placement on the east half and northern edge of the Site occurred between 1950 and 1952. Fill placement in the southwestern portion of the Site occurred between 1952 and 1960. By 1963 nearly the entire Site was paved and has remained largely unchanged since.

Project Scope:

The soil investigation will be completed by installing 22 test pits. The total depth of the test pits will be approximately 1 to 3 feet into native soil unless the capability of the excavotor is exceeded by depth or obstructions. One to three soil samples will be collected from each test pit to be determined by field conditions such as: total depth, visual observations, and photoionization detector readings. The spoils from the test pits will be collected on polyethelene sheeting and the open excavation and spoils will be photographed. The spoils will be returned to the excavation, compacted by the excavator, and the pavement will be replaced. The soil samples will be sent to the analytical laboratory for analysis of VOCs, SVOCs, and the RCRA 8 Metals.

The groundwater investigation will be completed by installing 22 shallow (up to 20 feet deep) and 2 deep wells (up to 35 feet deep). The monitoring wells will be installed in two rounds. The first round of wells will be installed, developed and sampled before installing the second round of wells to confirm if the locations are appropriate. The monitoring wells will be developed and sampled using low-flow methods for VOCs, SVOCs, and the RCRA 8 Metals.

Overview of Hazards:

Hazards expected to be encountered are the potential hazards related to the fill material, the heavy equipment to be used for investigation, buried or overhead utilities, fall hazards from the test pits utilized for investigation, and traffic.

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Site Status: Indicate current activity status and describe operations at the site.

☑ Active	☐ Inactive
☐ Partially active	☐ Other

The site is used for storage.

Site Plan:

Is a site plan or sketch available? ☑ Y ☐ N

Figure 3 from the investigation work plan it attached.

Work Areas:

List/identify each specific work area(s) on the job site and indicate its location(s) on the site plan:

1. Entire Site – Work will be conducted in all areas of the site.

Note: This HASP is developed for Haley & Aldrich purposes only and not for use by others.

3. PROJECT TASK BREAKDOWN

List and describe each distinct work task below.

Task No.	Detailed Task Description	Employee(s)	Work Date(s) or Duration
1	Soil Investigation – Test Pits	Enter employees	Enter dates/duration
2	Monitoring Well Installation	Enter employees	Enter dates/duration'
3	Well Development	Enter employees	Enter dates/duration'
4	Well Sampling	Enter employees	Enter dates/duration'

Note: This HASP is developed for Haley & Aldrich purposes only and not for use by others.

4. HAZARD ASSESSMENT

Material Safety Data Sheets (MSDS) of hazardous materials used during the execution of work shall be available on site. MSDSs are required for chemicals used to prepare samples, calibration gases, etc. MSDSs are not required for waste materials. MSDSs are available in Boston-based field vehicles and at the Roland Street Laboratory.

Chemical Hazards:

Does chemical analysis data indicate that the site is contaminated? ☑ Y ☐ N

Indicate the potential physical state of the hazardous materials at the site.

☑ Gas/Vapor	☑ Sludge
☑ Liquid	☑ Solid/Particulate

Indicate the anticipated or actual class of compounds at the site.

☐ Asbestos	☐ Inorganics
☑ BTEX	☐ Pesticides
☐ Chlorinated Solvents	☑ Petroleum products
☑ Heavy Metals	☑ Other VOCs

Impacted Environments:

Indicate media in which contamination is expected.

☐ Air	☑ Groundwater
☑ Soil	☐ Sediment
☐ Surface water	☐ Other Specify

Note: This HASP is developed for Haley & Aldrich purposes only and not for use by others.

Estimated concentrations:

The following chemicals have been detected at the site above Mo CALM Scenario C STARC levels.

Work Activity	Media	Chemical	Anticipated Concentration
Test Pit Sampling and Monitoring Well Installation	SO	Arsenic Beryllium Lead Mercury 1,2,4-Trimethylbenzene 1,3,5-Trimethylbenzene Benzo(a)pyrene Benzo(b)fluoranthene Di-n-octylphthalate Dibenzo(a,h)anthracene Naphthalene Total Extractable Hydrocarbons	20 mg/kg 1 mg/kg 14,000 mg/kg 2 mg/kg 1 mg/kg 250 mg/kg 2.7 mg/kg 1.5 mg/kg 1.1 mg/kg 0.72 mg/kg 430 mg/kg 65,000 mg/kg
Monitoring Well Sampling	GW	Arsenic Cadmium Iron Lead Manganese Chloroform Ethylbenzene n-Propylbenzene Toluene Xylene Bis(2-ethylhexel)phthalate	244 µg/l 7.6 µg/l 70,000 µg/l 31 µg/l 35,000 µg/l 230 µg/l 1,300 µg/l 1000,000 µg/l 54,000 µg/l 9,700 µg/l 47 µg/l

(Media key: A = Air; GW = Groundwater; SW = Surface Water; SO = Soil; SE = Sediment)

Chemicals of Concern:

Project action levels will be determined by the Haley & Aldrich Site Safety Officer based upon site conditions and information and will be presented in the task-specific health & safety requirements. The levels defined in Tables 1 and 2 of this HASP will serve as guidelines for project action levels.

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Chemicals of Concern:

Arsenic

The Occupational Safety and Health Administration has set limits of 10 microgram arsenic per cubic meter of workplace air (10 µg/m3) for 8 hour shifts and 40 hour work weeks. Several studies have shown that inorganic arsenic can increase the risk of lung cancer, skin cancer, bladder cancer, liver cancer, kidney cancer, and prostate cancer. The World Health Organization (WHO), the Department of Health and Human Services (DHHS), and the EPA have determined that inorganic arsenic is a human carcinogen.

Breathing high levels of inorganic arsenic can give you a sore throat or irritated lungs. Ingesting high levels of inorganic arsenic can result in death. Lower levels of arsenic can cause nausea and vomiting, decreased production of red and white blood cells, abnormal heart rhythm, damage to blood vessels, and a sensation of "pins and needles" in hands and feet.

Benzene

Benzene is a colorless liquid with a sweet odor. It evaporates into the air very quickly and dissolves slightly in water. It is highly flammable and is formed from both natural processes and human activities

Breathing very high levels of benzene can result in death, while high levels can cause drowsiness, dizziness, rapid heart rate, headaches, tremors, confusion, and unconsciousness. Eating or drinking foods containing high levels of benzene can cause vomiting, irritation of the stomach, dizziness, sleepiness, convulsions, rapid heart rate, and death. The major effect of benzene from long-term (365 days or longer) exposure is on the blood. Benzene causes harmful effects on the bone marrow and can cause a decrease in red blood cells leading to anemia. It can also cause excessive bleeding and can affect the immune system, increasing the chance for infection. Some women who breathed high levels of benzene for many months had irregular menstrual periods and a decrease in the size of their ovaries. It is not known whether benzene exposure affects the developing fetus in pregnant women or fertility in men. Animal studies have shown low birth weights, delayed bone formation, and bone marrow damage when pregnant animals breathed benzene.

Beryllium

Beryllium is a hard, grayish metal naturally found in mineral rocks, coal, soil, and volcanic dust. Beryllium compounds are commercially mined, and the beryllium is purified for use in nuclear weapons and reactors, aircraft and space vehicle structures, instruments, x-ray machines, and mirrors. Beryllium ores are used to make speciality ceramics for electrical and high-technology applications. Beryllium alloys are used in automobiles, computers, sports equipment (golf clubs and bicycle frames), and dental bridges. It enters water from erosion of rocks and soil, and from industrial waste. Some beryllium compounds will dissolve in water, but most stick to particles and settle to the bottom. Most beryllium in soil does not dissolve in water and remains bound to soil.

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Beryllium can be harmful if you breathe it. The effects depend on how much you are exposed to and for how long. If beryllium air levels are high enough (greater than 1000 µg/m^3), an acute condition can result. This condition resembles pneumonia and is called acute beryllium disease Occupational and community air standards are effective in preventing most acute lung damage.

BTEX

BTEX is the common abbreviation for benzene, toluene, ethyl benzene and xylene. OSHA has set permissible exposure limits for all of these contaminants that may be found at this worksite during your work activity. The levels that are set are based on an 8-hour time weighted average. Below are those values-

Benzene	1 ppm / 8 TWA
Toluene	200 ppm / 8 TWA
Ethyl Benzene	100 ppm / 8 TWA
Xylene	100 ppm / 8 TWA

Chloroform

Chloroform is a colorless liquid with a pleasant, nonirritating odor and a slightly sweet taste. It will burn only when it reaches very high temperatures.

In the past, chloroform was used as an inhaled anesthetic during surgery, but it isn't used that way today. Today, chloroform is used to make other chemicals and can also be formed in small amounts when chlorine is added to water.

Exposure to chloroform can occur when breathing contaminated air or when drinking or touching the substance or water containing it. Breathing chloroform can cause dizziness, fatigue, and headaches. Breathing chloroform or ingesting chloroform over long periods of time may damage your liver and kidneys. It can cause sores if large amounts touch your skin.

The Occupational Safety and Health Administration (OSHA) has set the maximum allowable concentration of chloroform in workroom air during an 8-hour workday in a 40-hour workweek at 50 ppm.

Di-n-octylphthalate

Di-*n*-octylphthalate is a colorless, odorless, oily liquid that doesn't evaporate easily. It is a man-made substance used to keep plastics soft or more flexible. This type of plastic can be used for medical tubing and blood storage bags, wire and cables, carpetback coating, floor tile, and adhesives. It is also used in cosmetics and pesticides. Di-*n*-octylphthalate sticks tightly to soil, sediment, and dust particles.

Little information is known about the health effects that might be caused by di-*n*-octylphthalate. It is not known what happens when you breathe or ingest the chemical. Some rats and mice that were given very high doses of di-*n*-octylphthalate by mouth died. Mildly harmful effects have been seen in the livers of some rats and mice given very high doses of di-*n*-octylphthalate by mouth for short (14 days or less) or intermediate periods (15 to 365 days) of time, but lower doses given for short periods of time generally caused no harmful effects.

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Lead

The Occupational Safety and Health Administration (OSHA) limits the concentration of lead in workroom air to 50 µg/cubic meter for an 8-hour workday. Lead can affect almost every organ and system in your body. The most sensitive is the central nervous system, particularly in children. Lead also damages kidneys and the immune system. The effects are the same whether it is breathed or swallowed. Exposure to lead is more dangerous for young and unborn children. Unborn children can be exposed to lead through their mothers. Harmful effects include premature births, smaller babies, decreased mental ability in the infant, learning difficulties, and reduced growth in young children. These effects are more common after exposure to high levels of lead. In adults, lead may decrease reaction time, cause weakness in fingers, wrists, or ankles, and possibly affect the memory. Lead may cause anemia, a disorder of the blood. It can cause abortion and damage the male reproductive system. The connection between these effects and exposure to low levels of lead is uncertain.

Manganese

Manganese is a naturally occurring metal that is found in many types of rocks. Pure manganese is silver-colored, but does not occur naturally. It combines with other substances such as oxygen, sulfur, or chlorine. Manganese occurs naturally in most foods and may be added to some foods.

Manganese is used principally in steel production to improve hardness, stiffness, and strength. It may also be used as an additive in gasoline to improve the octane rating of the gas. Manganese is routinely contained in groundwater, drinking water, and soil at low levels. Drinking water containing manganese or swimming or bathing in water containing manganese may expose you to low levels of this chemical.

The most common health problems in workers exposed to high levels of manganese involve the nervous system. These health effects include behavioral changes and other nervous system effects, which include movements that may become slow and clumsy. This combination of symptoms when sufficiently severe is referred to as "manganism". Other less severe nervous system effects such as slowed hand movements have been observed in some workers exposed to lower concentrations in the work place.

Workers exposed to high levels of airborne manganese in certain occupational settings may accumulate manganese dust on their work clothes. Manganese-contaminated work clothing should be removed before getting into your car or entering your home to help reduce the exposure hazard for yourself and your family.

Mercury

Mercury vapor is highly toxic via this route. Causes severe respiratory tract damage. Symptoms include sore throat, coughing, pain, tightness in chest, breathing difficulties, shortness of breath, headache, muscle weakness, anorexia, gastrointestinal disturbance, ringing in the ear, liver changes, fever, bronchitis and pneumonitis. Can be absorbed through inhalation with symptoms similar to ingestion. May cause burning of the mouth and pharynx, abdominal pain, vomiting, corrosive ulceration, bloody diarrhea. May be followed by a rapid and weak pulse, shallow breathing, paleness, exhaustion, tremors and collapse. Delayed death may occur from renal

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failure. Gastrointestinal uptake of mercury is less than 5% but its ability to penetrate tissues presents some hazard. Initial symptoms may be thirst, possible abdominal discomfort. Causes irritation and burns to skin. Symptoms include redness and pain. May cause skin allergy and sensitization. Can be absorbed through the skin with symptoms to parallel ingestion. Causes irritation and burns to eyes. Symptoms include redness, pain, and blurred vision; may cause serious and permanent eye damage. Chronic exposure through any route can produce central nervous system damage. May cause muscle tremors, personality and behavior changes, memory loss, metallic taste, loosening of the teeth, digestive disorders, skin rashes, brain damage and kidney damage. Can cause skin allergies and accumulate in the body. Repeated skin contact can cause the skin to turn gray in color. A suspected reproductive hazard; may damage the developing fetus and decrease fertility in males and females. Persons with nervous disorders, or impaired kidney or respiratory function, or a history of allergies or a known sensitization to mercury may be more susceptible to the effects of the substance.

The OSHA Acceptable Ceiling Concentration for mercury and mercury compounds: 0.1 mg/m3 (TWA), skin contact is a parameter in determining exposure for mercury.

Naphthalene

Exposure to naphthalene, 1-methylnaphthalene, or 2-methylnaphthalene happens mostly from breathing air contaminated from the burning of wood, tobacco, or fossil fuels, industrial discharges, or moth repellents. Exposure to large amounts of naphthalene may damage or destroy some of your red blood cells. Naphthalene has caused cancer in animals.

Naphthalene is a white solid that evaporates easily. Fuels such as petroleum and coal contain naphthalene. It is also called white tar, and tar camphor, and has been used in mothballs and moth flakes. Burning tobacco or wood produces naphthalene. It has a strong, but not unpleasant smell. The major commercial use of naphthalene is in the manufacture of polyvinyl chloride (PVC) plastics. Its major consumer use is in moth repellents and toilet deodorant blocks.

Exposure to large amounts of naphthalene may damage or destroy some of your red blood cells. This could cause you to have too few red blood cells until your body replaces the destroyed cells. This condition is called hemolytic anemia. Some symptoms of hemolytic anemia are fatigue, lack of appetite, restlessness, and pale skin. Exposure to large amounts of naphthalene may also cause nausea, vomiting, diarrhea, blood in the urine, and a yellow color to the skin. Animals sometimes develop cloudiness in their eyes after swallowing high amounts of naphthalene. It is not clear whether this also develops in people. Rats and mice that breathed naphthalene vapors daily for a lifetime developed irritation and inflammation of their nose and lungs. It is unclear if naphthalene causes reproductive effects in animals; most evidence says it does not.

The Occupational Safety and Health Administration (OSHA) set a limit of 10 ppm for the level of naphthalene in workplace air during an 8-hour workday, 40-hour workweek. The National Institute for Occupational Safety and Health (NIOSH) considers more than 500 ppm of naphthalene in air to be immediately dangerous to life or health. This is the exposure level of a chemical that is likely to impair a worker's ability to leave a contaminate area and therefore, results in permanent health problems or death.

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Polycyclic Aromatic Hydrocarbons

Polycyclic aromatic hydrocarbons (PAHs) are a group of over 100 different chemicals that are formed during the incomplete burning of coal, oil and gas, garbage, or other organic substances like tobacco or charbroiled meat. PAHs are usually found as a mixture containing two or more of these compounds, such as soot.

Some PAHs are manufactured. These pure PAHs usually exist as colorless, white, or pale yellow-green solids. PAHs are found in coal tar, crude oil, creosote, and roofing tar, but a few are used in medicines or to make dyes, plastics, and pesticides. In soils, PAHs are most likely to stick tightly to particles; certain PAHs move through soil to contaminate underground water.

The Occupational Safety and Health Administration (OSHA) has set a limit of 0.2 milligrams of PAHs per cubic meter of air (0.2 mg/m^3). The OSHA Permissible Exposure Limit (PEL) for mineral oil mist that contains PAHs is 5 mg/m^3 averaged over an 8-hour exposure period.

Toluene

Toluene affects the brain. Low-to-moderate levels from long-term exposure can cause tiredness, confusion, weakness, drunken-type actions, memory loss, nausea and loss of appetite, and hearing loss. Inhaling a high level of toluene in a short time can make you feel light-headed, dizzy, or sleepy. It can cause unconsciousness, and even death. Repeated exposure to high levels can cause permanent brain and speech damage, vision and hearing problems, loss of muscle control, and poor balance. It can also cause memory loss and decreased mental ability. Toluene also affects the kidneys. Several studies have shown that unborn animals were harmed when high levels of toluene were breathed by their mothers. Babies can have neurological problems and retarded growth and development if their mothers.

Total Petroleum Hydrocarbons

Total petroleum hydrocarbons (TPH) is a term used to describe a large family of several hundred chemical compounds that originally come from crude oil. Crude oil is used to make petroleum products, which can contaminate the environment. Because there are so many different chemicals in crude oil and in other petroleum products, it is not practical to measure each one separately. However, it is useful to measure the total amount of TPH at a site. TPH is a mixture of chemicals, but they are all made mainly from hydrogen and carbon, called hydrocarbons. Scientists divide TPH into groups of petroleum hydrocarbons that act alike in soil or water. These groups are called petroleum hydrocarbon fractions. Each fraction contains many individual chemicals. Some chemicals that may be found in TPH are hexane, jet fuels, mineral oils, benzene, toluene, xylenes, naphthalene, and fluorene, as well as other petroleum products and gasoline components. However, it is likely that samples of TPH will contain only some, or a mixture, of these chemicals.

Some of the TPH compounds can affect your central nervous system. One compound can cause headaches and dizziness at high levels in the air. Another compound can cause a nerve disorder called "peripheral neuropathy," consisting of numbness in the feet and legs. Other TPH compounds can cause effects on the blood, immune system, lungs, skin, and eyes. Animal studies have shown effects on the lungs, central nervous system, liver, and kidney from

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exposure to TPH compounds. Some TPH compounds have also been shown to affect reproduction and the developing fetus in animals.

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(CIRCLE CONTAMINANTS OF CONCERN, WRITE ADDITIONAL CONTAMINANTS AND EXPOSURE ON LAST PAGE)

CHEMICAL	ROUTES OF EXPOSURE	IDLH	Ceiling	STEL	PEL	TLV	REL	PID (IP eV)	FID	ODOR THRES-HOLD	IRRITATION THRESHOLD	ODOR DESCRIPTION
VAPORS & GASES												
Acetone	R, I, C	2500	-	750 [ACGIH]	1000	500	250	9.69	60	13	-	fragrent, mint-like
Ammonia	R, I, C	300	-	35 [NIOSH, ACGIH]	50	25	25	10.18**	-	0.5-2	10	Pungent suffocating odor
Benzene	R,A,I,C	Ca [500]	-	1 [NIOSH]; 2.5 [ACGIH]	1	0.5	0.1	9.24	150	4.68	-	Solvent, aromatic
Carbon tetrachloride (Tetrachloromethane)	R,A,I,C	Ca [200]	25 [instantaneous] 200 [5 min peak in any 4 hrs]	2 [NIOSH, 60-min]; 10 [ACGIH]	2	5	Ca	11.47**	10	50	-	Sweet, pungent, ether-like
Chlorobenzene	R,I,C	1000	-	-	75	10	-	9.07	200	0.68	-	Almond-like
Chloroform	R,I,C	Ca [500]	50 [OSHA]	2 [NIOSH, 60-min]	-	10	-	11.42**	65	50	-	Sweet, pleasant
o-Dichlorobenzene	R,A,I,C	200	50 [NIOSH, OSHA]	50 [ACGIH]	-	25	-	9.06	50	0.3	E 20-30	Pleasant, aromatic
p-Dichlorobenzene	R,A,I,C	Ca [150]	-	-	75	10	Ca	8.98	-	0.18	E 80-160	Distinct, aromatic, mothball-like
Dichlorodifluoromethane (Freon 12)	R,C	15000	-	-	1000	1000	1000	11.75**	15	-	-	Ether-like when at very high concs.
1,1-Dichloroethane	R,I,C	3000	-	-	100	100	100	11.06**	80	200	-	Distinct, chloroform-like
1,2-Dichloroethane (Ethylene dichloride)	R,I,A,C	Ca [50]	100 [OSHA]	2 ppm [NIOSH]; 200 ppm [OSHA, 5-min max peak in any 3 hours]	50	10	1	11.05**	80	88	-	Chloroform-like
1,1-Dichloroethylene (1,1-DCE, Vinylidene chloride)	R,A,I,C	Ca [ND]	-	-	-	5	Ca	10.00**	40	190	-	Chloroform-like
1,2-Dichloroethylene	R,I,C	1000	-	-	200	200	200	9.65	50	0.85	-	Bitter, chloroform-like
Ethanol	R,I,C	3300	-	-	1000	1000	1000	10.47**	25	10	-	Weak, ether-like, wine-like
Ethylbenzene	R,I,C	800	-	125 [NIOSH; ACGIH]	100	100	100	8.76	100	2.3	E 200	Aromatic
Ethylene Glycol	R,I,C	ND	50 [OSHA]; 100 mg/m^3 [ACGIH]	-	-	-	-	-	-	-	-	Odorless
Formaldehyde	I,C	Ca [20]	0.1 [NIOSH, 15-min]; 0.3 [ACGIH]	2	0.75	-	Ca [0.016]	10.88**	-	0.83	-	Pungent, suffocating
Gasoline	R,I,A,C	Ca [ND]	-	500 [OSHA; ACGIH]	300	300	-	-	-	-	E 0.5	Petroleum-like
n-Hexane	R,I,C	1100	-	-	500	50	50	10.18	70	130	E.T 1400-1500	Gasoline-like
Hydrogen Cyanide	R,A,I,C	50	4.7 [ACGIH; Skin]	4.7 [NIOSH - skin]	10 [skin]		-	-	-	0.58	-	Bitter almond
Hydrogen peroxide	R,I,C	75	-	-	1	1	1	10.54**	-	-	-	Sharp
Methanol	R,I,A,C	6000	-	250 [NIOSH; ACGIH; skin]	200	200 [skin]	200	10.84**	12	1000	-	Pungent
Methyl Ethyl Ketone Peroxide	R,I,C	ND	0.2 [NIOSH; ACGIH] 0.7 [OSHA]	-	-	-	-	-	-	-	-	Characteristic odor
Methyl Chloroform (1,1,1-TCA)	R,I,C	700	350 [NIOSH, 15-min]	450 [ACGIH]	350	350	Ca	11.00**	105	20-100	-	Chloroform-like
Methylene Chloride (Dichloromethane, Methylene dichloride)	R,I,A,C	Ca [2300]	-	125	25	50	Ca	11.32**	100	25-50	E 5000	Chloroform-like
Methyl Mercaptan	R,C	150	10 [OSHA] 0.5 [NIOSH, 15-min]	-	-	0.5	-	9.44	-	-	-	Garlic, rotten cabbage
MIBK (Hexone)	R,I,C	500	-	75 [NIOSH; ACGIH]	100	50	50	9.30	-	-	-	Pleasant
Naptha (coal tar)	R,I,C	1000	-	-	100	400	100	-	-	-	-	Aromatic
Naphthalene	R,A,I,C	250	-	15 [NIOSH; ACGIH]	10	10	10	8.12	-	0.3	E 15	Mothball-like
Octane	R,I,C	1000	385 [NIOSH, 15-min]	-	500	300	75	9.82	80	48	-	Gasoline-like
Pentachlorophenol	R,A,I,C	2.5 mg/m^3	-	-	0.5 mg/m^3 [skin]	0.5 mg/m^3 [skin]	0.5 mg/m^3 [skin]	-	-	-	-	Pungent when hot, benzene-like
Phenol	R,A,I,C	250	15.6 [NIOSH, 15-min]	-	5 [skin]	5 [skin]	5 [skin]	8.50	-	0.04	E.N.T. 68	Sweet, acrid
Propane	R,C	2100	-	-	1000	1000	1000	11.07**	80	1600	-	Odorless (commonly smells foul due to additive for odor detection)
Stoddard Solvent (Mineral Sprits)	R,Cl,I	20000 mg/m^3	1800 mg/m^3 [NIOSH, 15-min]	-	500	100	350 mg/m^3	-	-	1	E 400	Kerosene-like
Styrene	R,I,A,C	700	200 [OSHA]	100 [NIOSH]; 600 [OSHA, 5-min max peak in any 3 hours]; 40 [ACGIH]	100	20	50	8.40	85	0.047	E 200-400	Sweet, floral
1,1,2,2-Tetrachloroethane	R,I,A,C	Ca [100]	-	-	5 [skin]	1 [skin]	1 [skin]	11.10**	100	1.5	-	Pungent, chloroform-like
Tetrachloroethylene (Perchloroethylene, Perc, PCE)	R,I,A,C	Ca [150]	200 [OSHA]	300 [OSHA, 5-min max peak in any 3-hours]; 100 [ACGIH]	100	25	Ca	9.32	70	4.68	N.T513-690	Chloroform-like
Toluene	R,A,I,C	500	300 [OSHA]	150 [NIOSH]; 500 [OSHA, 10-min max peak]	200	50	100	8.82	110	2.14	E300-400	Sweet, pungent, benzene-like
Trichloroethylene (TCE)	R,I,A,C	Ca [1000]	200 [OSHA]	300 [OSHA, 5-min max peak in any 2-hours]; 100 [ACGIH]	100	50	Ca	9.45	70	21.4	-	Chloroform-like
1,2,3-Trimethylbenzene	R,I,C	ND	-	-	-	-	25	8.48	-	-	-	Distinctive, aromatic
1,2,4-Trimethylbenzene	R,I,C	ND	-	-	-	-	25	8.27	-	-	-	Distinctive, aromatic
1,3,5-Trimethylbenzene	R,I,C	ND	-	-	-	-	25	8.39	-	-	-	Distinctive, aromatic
Turpentine	R,A,I,C	800	-	-	100	20	100	-	-	200	E.N 200	Pine-like
Vinyl Chloride	R,C	Ca [ND]	5 [OSHA, 15-min]	-	1	1	Ca	9.99	-	3000	-	Pleasant odor at high concs.
Xylenes	R,A,I,C	900	-	150 [NIOSH, ACGIH]	100	100	100	8.56 (m- and o-) 8.44 (p-)	111/116	1.1	E.N.T. 200	Aromatic

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(CIRCLE CONTAMINANTS OF CONCERN, WRITE ADDITIONAL CONTAMINANTS AND EXPOSURE ON LAST PAGE)

CHEMICAL	ROUTES OF EXPOSURE	IDLH	Ceiling	STEL	PEL	TLV	REL	PID (IP eV)	FID	ODOR THRES-HOLD	IRRITATION THRESHOLD	ODOR DESCRIPTION
DUSTS, MISTS, FUMES, AND MISCELLANEOUS COMPOUNDS												
Asbestos	R	Ca (ND)	-	-	0.1 fiber/cc	0.1 fiber/cc	0.1 fiber/cc	-	-	-	-	-
PCBs-42% Chlorine	R,A,I,C	Ca [5 mg/m^3]	-	-	1 mg/m^3 [skin]	1 mg/m^3 [skin]	0.001 mg/m^3	-	-	-	-	Mild, hydrocarbon
PCBs-54% Chlorine	R,A,I,C	Ca [5 mg/m^3]	-	-	0.5 mg/m^3 [skin]	0.5 mg/m^3 [skin]	0.001 mg/m^3	-	-	-	-	Mild, hydrocarbon
Aluminum - metal dust	R,C	ND	-	-	15 mg/m^3 (total); 5 mg/m^3 (respirable)	10 mg/m^3	10 mg/m^3 (total), 5 mg/m^3 (respirable)	-	-	-	-	-
Aluminum - soluble salts	R,I,C	ND	-	-	2 mg/m^3	2 mg/m^3	2 mg/m^3	-	-	-	-	-
Arsenic- inorganic	R,A,I,C	Ca [5 mg/m^3]	0.002 mg/m^3 [NIOSH, 15-min]	-	0.01 mg/m^3	0.01 mg/m^3	Ca	-	-	-	-	-
Barium:soluble compounds	R,I,C	50 mg/m^3	-	-	0.5 mg/m^3	0.5 mg/m^3	0.5 mg/m^3	-	-	-	-	-
Beryllium	R,C	Ca [4 mg/m^3]	0.005 mg/m^3 [OSHA]; 0.025 mg/m^3 [OSHA, 30-min max peak]; 0.0005 mg/m^3	0.01 mg/m^3 [ACGIH]	0.002 mg/m^3	0.002 mg/m^3	Ca	-	-	-	-	-
Cadmium dusts	R,I	Ca [9 mg/m^3]	-	-	0.005 mg/m^3	0.01 mg/m^3	Ca	-	-	-	-	-
Chromates (Cr(VI) Compounds) & Chromic Acid	R,I,C	Ca [15 mg/m^3]	0.1 mg/m^3 [OSHA]	-	0.001 mg/m^3	0.05 mg/m^3 [water soluble]; 0.01 mg/m^3 [insoluble]	Ca	-	-	-	-	-
Chromium (III) Compounds	R,I,C	25 mg/m^3	-	-	0.5 mg/m^3	0.5 mg/m^3	0.5 mg/m^3	-	-	-	-	-
Chromium Metal	R,I,C	250 mg/m^3	-	-	1 mg/m^3	0.5 mg/m^3	0.5 mg/m^3	-	-	-	-	-
Copper - dust & mist	R,I,C	100 mg/m^3	-	-	1 mg/m^3	1 mg/m^3	1 mg/m^3	-	-	-	-	-
Lead	R,I,C	100 mg/m^3	-	-	0.050 mg/m^3	0.05 mg/m^3	0.050 mg/m^3	-	-	-	-	-
Manganese (compounds and fume)	R,I	500 mg/m^3	5 mg/m^3 [OSHA]	3 mg/m^3 [NIOSH]	-	0.2 mg/m^3	1 mg/m^3	-	-	-	-	-
Mercury & Inorganic Mercury Compounds	R,I,A,C	10 mg/m^3	0.1 mg/m^3 [NIOSH, Skin]; 0.1 mg/m^3 [OSHA]	-	-	0.025 mg/m^3	0.05 mg/m^3 [skin]	-	-	-	-	-
Organo-Mercury Compounds	R,A,I,C	2 mg/m^3	0.04 mg/m^3 [OSHA]	0.03 mg/m^3 [NIOSH]	0.01 mg/m^3	0.01 mg/m^3 [alkyl]; 0.1 mg/m^3 [aryl]	0.01 mg/m^3	-	-	-	-	-
Nickel (metal and compounds)	R,I,C	Ca [10 mg/m^3]	-	-	1 mg/m^3	1.5 mg/m^3 [metal]; 1 mg/m^3 [soluble inorganic compounds]; 1 mg/m^3 [insoluble	0.015 mg/m^3	-	-	-	-	-
Particulate (Not otherwise regulated)	R, C	ND	-	-	15 mg/m^3 (total); 5 mg/m^3 (respirable)	10 mg/m^3 (inhalable); 3 mg/m^3 (respirable)	-	-	-	-	-	-
Portland cement	R,I,C	5000 mg/m^3	-	-	50 mppcf	10 mg/m^3	10 mg/m^3 (total); 5 mg/m^3 (respirable)	-	-	-	-	-
Selenium compounds	R,I,C	1 mg/m^3	-	-	0.2 mg/m^3	0.2 mg/m^3	0.2 mg/m^3	-	-	-	-	-
Silica, crystalline	R, C	Ca [25 mg/m^3 (cristobalite, tridymite); 50 mg/m^3 (quartz, tripoli)]	-	-	Dependent on silicon dioxide content of silica (see Appendix C of the NIOSH Pocket Guide to Chemical Hazards, 2000)	Dependent on minerology [see ACGIH 2005 TLVs and BEIs Handbook]	0.05 mg/m^3	-	-	-	-	-
Silver (metal and soluble compounds)	R,I,C	10 mg/m^3	-	-	0.01 mg/m^3	0.1 mg/m^3	0.01 mg/m^3	-	-	-	-	-
Thallium, soluble	R,A,I,C	15 mg/m^3	-	-	0.1 mg/m^3 [skin]	0.1 mg/m^3 [skin]	0.1 mg/m^3 [skin]	-	-	-	-	-
Tin (metal)	R,C	100 mg/m^3	-	-	2 mg/m^3	2	2 mg/m^3	-	-	-	-	-
Tin (organic compounds)	R,A,I,C	25 mg/m^3	-	-	0.1 mg/m^3	0.1 mg/m^3 [skin]	0.1 mg/m^3 [skin]	-	-	-	-	-
Zinc oxide dust & fume	R	500 mg/m^3	15 mg/m^3 [NIOSH, dust]	10 mg/m^3 [NIOSH; ACGIH, fume]	15 mg/m^3 (total dust), 5 mg/m^3 [respirable dust]; 5 mg/m^3 [fume]	2 mg/m^3 [respirable]	5 mg/m^3 (total dust), 5 mg/m^3 [fume]	-	-	-	-	-

NOTES & ABBREVIATIONS:

All units in parts per million (ppm) unless otherwise noted.
R = Respiratory (Inhalation)
I = Ingestion
A = Skin Absorption
C = Skin Contact
-: Not available
ND: Not detectable.
Ca = Carcinogen
** = Use 11.7 eV lamp
IP: Ionization potential
eV: Electrovolts

IDLH: Immediately dangerous to life and health
Ceiling: Highest allowable instantaneous
C = Skin and/or Eye Contact
STEL: Short-term exposure limit. Exposure period is 15 minutes unless otherwise indicated
PEL: OSHA Permissible Exposure Limit (legally-enforceable)
REL: NIOSH Recommended Exposure Limit
PID: Photoionization Detector
OSHA: United States Occupational Safety and Health Administration
NIOSH: National Institute of Occupational Safety and Health
TLV: ACGIH Threshold Limit Value
ACGIH: American Conference of Governmental Industrial Hygienists

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Physical Hazards:

Indicate all hazards that may be present for each task. If any of these potential hazards are checked, it is the project manager's responsibility to determine how to eliminate/minimize the hazard to protect onsite personnel.

Copy and paste a checkmark "✓"into appropriate boxes.

Physical Hazard Checklist				
Potential Job Hazards	**Task 1**	**Task 2**	**Task 3**	**Task 4**
	Test Pits	MW Install	Well Development	Well Sampling
Confined space entry*				
Underground utilities	✓	✓		
Overhead utilities	✓	✓		
Electrical hazards				
Excavations greater than 4' depth	✓			
Open excavation fall hazards	✓			
Heavy equipment	✓	✓		
Drilling hazards		✓		
Noise (above 85 dBA)	✓	✓		
Traffic concerns	✓	✓	✓	✓
Extreme weather conditions	✓	✓	✓	✓
Rough terrain for drilling equipment				
Buried drums				
Heavy lifting (more than 50 lbs)	✓	✓	✓	✓
High risk fire hazard				
Poisonous insects or plants				
Water hazards				
Use of a boat				
Lockout/Tagout requirements				
Other: Specify				

***CONFINED SPACE ENTRY REQUIRES SPECIAL PROCEDURES, PERMITS AND TRAINING AND MUST BE APPROVED BY THE CORPORATE HEALTH & SAFETY MANAGER.**

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Potential Activity Hazards and Hazard Controls:

Copy and paste a checkmark "✓" adjacent to potential activity hazards and relevant hazard controls.

POTENTIAL ACTIVITY HAZARDS

Abrasions and Cuts ✓
Access
Asphyxiation
Bacteria
Biological Hazards
Bloodborne Pathogens
Cave Ins
Chemical/Thermal Burns
Chemicals
Cold Stress ✓
Compressed Gases
Confined Spaces
Congestion
Defective Equipment
Dermatitis
Dropping Materials/Tools to Lower Levels
Drowning or Flowing Water
Electrical Shock
Energized Equipment
Equipment Misuse ✓
Ergonomics
Excavations ✓
Explosions
Fatigue
Fire ✓
Flammability
Flying debris ✓
Foreign Body in Eye ✓
Frostbite/Cold
Fueling and Fuel Storage ✓
Fugitive Dust ✓
Fumes ✓
Generated Wastes
Guards removed
Hazardous Materials ✓
Heat Stress (cramps, exhaustion, stroke)
Heavy Equipment Operation ✓
Heavy Equipment/Stability ✓
Heavy Lifting ✓
High crime area (violence)
High Winds
Hoists, Rigging, Slings, Cables ✓
Housekeeping – Improper ✓ Illumination - Poor
Impact ✓
Inability to Maintain Communication
Inclement Weather ✓
Inclines
Insects/Reptiles
Mold
Moving Equipment, Conveyors or Vehicles ✓
Muddy Site Conditions
New Personnel
Noise ✓
Odor ✓
Overhead Utilities ✓
Overhead Work
Overloaded Equipment
Oxygen deficiency
Pinch Points ✓
Poisonous Plants
Pressure
Pressurized Lines ✓
Radiation
Repetitive Motion
Rigging - Improper ✓
Sharp Objects ✓
Silicosis
Slips, Trips, and Falls ✓
Sprains and Strains ✓
Steam
Sunburn
Surface Water Run-off
Toxicity ✓
Traffic ✓
Underground Utilities ✓
Uneven Terrain
Unsafe Atmosphere
Vibration
Visibility - Poor
Visitors Known/Unknown
VOC Emissions ✓
Weight ✓
Work at Depth
Work at Heights
Work over Water
Working on Ice

HAZARD CONTROLS

Air Monitoring ✓
Appropriate Clothing/Monitoring Of Weather ✓
Appropriate Labels/Signage ✓
Barricades/Fencing/Silt Fencing ✓
Buddy System - Attendant
Chock Blocks ✓
Confined Space Procedures
Decontamination Procedures ✓
Derived Waste Management Plan
Drinking Water/Fluids ✓
Dust Abatement Measures ✓
Emergency Action Plan Procedures
Equipment Inspection ✓
Equipment Manuals/Training
Exclusion/Work Zones ✓
Exhaust Ventilation
Eye Protection ✓
Fall Protection
Fire Extinguisher ✓
Flotation Devices/Lifelines
Gloves ✓
Ground Fault Interrupter
Grounded Hydraulic Attachments
Grounded Equipment/Tanks
Hand Signal Communication✓
Hard Hat ✓
Hazardous/Flammable Material Storage
Hearing Protection ✓
High Visibility Safety Vest ✓
Hoses, Access to Water
Hotwork Procedures
Isolation of Energy Sources(Lockout/Tagout)
Machine/Equipment Guards
Manual Lifting Equipment
Police Detail ✓
Proper Lifting Techniques ✓
Proper Tool for Job ✓
Proper Work Position/Tools ✓
Protective Equipment ✓
Radio Communication
Respirator, (Specify Type)
Safety Harness /Lanyard/Scaffold
Security Escort
Sloping, Shoring, Trench Box
Spill Prevention Measures
Spill Kits
Stormwater Control
Traffic Controls ✓
Procedures/Methods
Vehicle Inspection ✓
Visitor Orientation Escort
Window Cleaning/Defrost ✓

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Specific Activity Hazards and Precautions

None

Safety Meetings

All H&A personnel visiting the site will be given an orientation safety meeting and are required to read and sign this HASP. Daily safety meetings will be conducted onsite and documented on a Health & Safety Tailgate Meeting Form.

Utility Locators and Underground Hazards

Prior to drilling, Haley & Aldrich staff members will ensure that permission has been gained from the property owner to access the property. Before marking any proposed exploration or drilling location, it is critical that all readily available information on underground utilities and structures be obtained. The estimated location of utility installations, such as sewer, telephone, fuel, electric, water, or any other underground installation that may be expected to be encountered during drilling work, will be identified with the appropriate authority. Appropriate authorities include client representatives, utility companies, nonprofit organizations (e.g., "Dig-Safe), and others. A list of all state “utility locators” is posted on the Health and Safety Homepage under “Guidance Documents”.

Note: It is important to note that not all utilities are participants in the “one-call” agency or process. As such, inquiries must be made with the “one-call” agency to determine which entities do not participate, so they can be contacted independently.

Also, most stake-outs or markings have a limited time period for which they remain valid, typically 2 to 3 weeks. It is critical that this time period be taken into account to prevent expiration of clearance prior to completion of the invasive activities, and the need to repeat the process.

Completion of the utility stake out is not a guarantee that the underground facilities will not be encountered in the boreholes; Very few if any guarantee their work nor do they accept the liability for damage or losses if one may occur. Accordingly, Haley & Aldrich field staff are expected to use extreme caution in the upper 4-5 feet in the event the clearance has failed to identify an existing facility. This may necessitate hand-excavation or probing to confirm the presence of shallow utilities.

When utility companies or owners cannot respond to a request to locate underground utility installations within 24 hours (unless a longer period is required by state or local law), or cannot establish the exact location of these installations, geophysical techniques, such as ground penetrating radar and/or magnetometery can be utilized to locate the potential underground hazards. Using any information that can be obtained, the site should be viewed in detail for physical evidence of buried lines or structures. Evidence of surface elements of buried utilities should be documented, such as manholes, gas or water valves, catch basins, etc.

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No subsurface drilling activities will be allowed until "all" utilities have been properly located and marked.

Marking locations can be accomplished using spray paint on the ground, stakes, or other similar method. All markings of proposed locations shall be made in white, in accordance with the generally-accepted universal color code for facilities identification (AWMA 4/99).

White:	Proposed excavation or drilling location
Pink:	Temporary Survey Markings
Red:	Electrical, Power Lines, Cables, Conduit, and Lightening Cables.
Yellow:	Gas, Oil, Steam, Petroleum , and Gaseous Materials.
Orange:	Communications, Alarm, or Signal Lines, Cables, and Conduits.
Blue:	Potable Water.
Purple:	Reclaimed Water, Irrigation, and Slurry Lines.
Green:	Sewers and Drain Lines.

The public and private utility entities generally only mark the locations of their respective underground facilities within public rights-of-way. Determination of the locations on private property will most likely be the responsibility of Haley & Aldrich or the contractor. In some cases, it may be necessary to put the ultimate responsibility back on the owner, to assist in the location of the utilities. It is incumbent on Haley & Aldrich and the Contractor to exercise caution and use good judgement when faced with uncertainty.

Excavation and Trenching Safety

The following is a list of minimum requirements for trenching and excavating. Each excavation/trench/shoring project is different, therefore the Contractor/Consultant Project Manager is responsible for evaluating site specific conditions and making appropriate provisions in the task-specific health and safety requirements in conformance with 29 CFR 1926 Subpart P - Excavations.

- Contact the proper utilities to obtain clearance. Prior to work, review the utilities in the area and be sure they have been staked properly. Before work begins, a Safe Work Permit must be obtained from the Client/Site manager of Operations Safety Representative.

- Be aware that trenches and excavations deeper than four feet are considered confined spaces and require additional safety precautions, such as shoring. If an excavation exceeds four feet in depth, contact the Client or H&A Safety Representative to review the original Safe Work Permit and ensure that it is adequate.

- The walls and faces of all excavations and trenches more than four feet deep, in which an staff member is exposed to danger from moving ground, will be guarded by a shoring system, sloping of the ground, or some other equivalent means. The design of shoring systems must be done by a registered Professional Engineer as per 29 CFR 1926 Subpart P.

- Staff members are not allowed to enter test pits. Sample collection will be performed with the bucket of the excavator.

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- Hard hats and other personal protective equipment will be worn at all times during any type of excavating or trenching operation.

- Determine soil composition (e.g., through soil sampling, soil maps, etc.) and other relevant site conditions, with special emphasis on conditions conducive to cave-ins.

- Monitor the atmosphere in and around trenches on a regular basis to check for explosive, toxic or otherwise dangerous gases and vapors.

- The Contractor/Consultant Project Manager will insure that all staff members involved in the excavation activity have appropriate training in safe trenching practices, with emphasis on factors such as:
    - utility line identification
    - cave-in prevention measures
    - recognition of conditions which may cause cave-ins

- Water will not be allowed to accumulate in any excavation. Utilize ditches, dikes, pumps, or other means to keep surface water out of trenches.

- All open excavations must be well marked and barricaded.

Excavation Cave-In Hazards

- The following conditions increase the likelihood of cave-in: Soil materials composed of unconsolidated, uncompacted, and/or rounded particles (See 29 CFR 1926 Subpart P - Excavation Standard). Special care must be used when trenching in areas that have previously been excavated and backfilled.

- Soils, which have a high water, content, or have been subjected to freeze-thaw or frost-heaving.

- Loading of trench walls by adjacent equipment, supplies, structures, "back-dirt" piles, etc.

- Vibration due to equipment operating near excavations.

- Trench walls that are steeper than the angle of repose of the material composing the walls.

- Deep trenches (i.e., high trench walls).

    The following precautions should be used to prevent cave-ins in all trenches in excess of 4 ft. deep. These precautions should also be used in trenches less than 4 ft. deep whenever those site conditions just listed indicate the likelihood of a cave-in:

    - Sloping: Trench walls should be sloped to the correct angle of repose.

    - Shoring: Vertical trench walls (unless composed of solid rock) must be shored and braced, or restrained with movable trench boxes, to prevent cave-in. A registered

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professional engineer must design shoring systems and meet accepted engineering requirements.

Heavy Equipment

Staff Members must be especially careful and alert when working with contractors who use heavy equipment, since equipment failure or breakage can lead to accidents and worker injury. Cranes and equipment for drilling, pile driving, test pitting and coring is of special concern. Should these devices fail during operation the likelihood of worker injury is high. Equipment of this nature should be visually inspected and checked for proper working order prior to the commencement of field work. Those that operate heavy equipment must meet all of the requirements to operate heavy equipment. Haley & Aldrich, Inc. staff members that supervise projects or are associated with such high risk projects that involve digging should use due diligence when working with a construction firm. Maintain visual contact with operators at all times and keep out of the strike zone whenever possible. Always approach heavy equipment with an awareness of the swing radius and traffic routes of each piece of equipment and never go beneath a hoisted load. High-visibility safety vests must be worn onsite at all times. Avoid fumes created by heavy equipment exhaust.

Rig Inspection

Each day, prior to the start of work, the driller will inspect the drill rig and associated equipment. The following checks will be made:

- Vehicle condition: Check proper operation of brakes, lights, steering mechanism, and horn.
- Equipment storage: All equipment such as auger flights, split spoon samplers, hammers, hand tools, etc. will be properly stored in an appropriate location and will be secured before moving the rig.
- Wire rope, Cat Line: All wire rope, cable and Cat Line will be inspected for signs of wear such as broken wires, a reduction in rope diameter, abrasion, or signs of rust. Worn, frayed, or otherwise damaged wire, rope or cable will be replaced.
- Safety equipment: Each rig will have at least one fire extinguisher (Type B/C) and one First Aid Kit.

Rig Set-Up

Each drill rig will be properly blocked and leveled prior to raising the derrick. The rig will be moved only after the derrick has been lowered. The leveling jacks will not be raised until the derrick has been lowered.

Blocking provides a more stable drilling structure by evenly distributing the weight of the rig. Proper blocking ensures that a differential settling of the rig does not occur. Wooden blocks, at least 12 by 12 inches and four to eight inches thick, are recommended and should be placed between the jack swivels and the ground. The emergency brake will be engaged and the wheels that are on the ground chocked.

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Site drilling will comply with the following rules:

- Before drilling, the Contractor/Consultant Site Coordinator will ensure an adequate safety zone around the drill rig and associated operations.

- Before drilling, the existence of underground utilities in the work area will be determined and conspicuously marked.

- If drilling is conducted in the vicinity of overhead power lines, proper distance will be maintained between the drill rig and the lines as per OSHA 29 CFR 1926, Subpart N. The proper distance or shielding technique will be stated in the project-specific HASP.

Noise Reduction

Site activities in proximity to heavy equipment often expose workers to excessive noise. It is anticipated that situations may arise when noise levels may exceed the OSHA Action Level of 85 dBA in an 8-hour time-weighted average (TWA). An example of this possibility is working in close proximity to the subcontractor during drilling activities onsite. If excessive noise levels occur, efforts will be made to control this by issuance of earplugs to all personnel and by implementing a system of hand signals understood by all.

Work Site Access & Controls (Standard Precautions)

The work area is restricted to authorized personnel. Clearly define the work area before beginning activities for the day. Caution tape and safety cones must be provided as necessary for vehicular traffic concerns and to protect passers-by. Proper housekeeping is essential to avoid creating hazards to pedestrian and vehicular traffic. Excavations in progress will not be left unattended at any time. Running equipment will not be left unattended at any time. Test borings and test pits will be backfilled upon completion and the area restored. Drilling equipment will be secured above test borings during work stoppages and at the end of the workday.

Site Security

Site is fenced.

Weather Related Hazards

H&A employees and their subcontractors should be aware of potential health effects and/or physical hazards of working during inclement weather. Refer to OP1003-Cold Stress and OP1015-Heat Stress for discussion on weather hazards.

Lightning

- Always pay attention to the weather conditions. You are responsible for your own safety. Use common sense and do not feel pressure to continue to work if you feel there is a threat and others don't, such as contractors and co-workers.

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- If you are using conductive tools and equipment, separate yourself from them as far as practical.
- If you are near a drilling rig, lower the mast and move away from the rig.
- Rule of thumb- wait until 30 minutes after the last observed lightning strike or thunderclap before resuming your outdoor activities, warns the National Lightning Safety Institute.
- Protect yourself by taking cover in the best shelter you can find.
- If you are in or near the water, go to land immediately and find shelter. Take extra precaution when on the water and in a boat.
- If choosing between a building and a car, choose the building.
- If choosing between a hardtop and a convertible, choose the hardtop.
- If you're in a car, keep the windows closed.
- If there is no shelter, find a low-lying, open place that is a safe distance from trees, poles, or metal objects that can conduct electricity. Make sure it is not likely to flood.
  Assume a tucked position: Squat low to the ground. Place your hands on your knees with your head tucked between them. Try to touch as little of your body to the ground as possible.
- Do not lie flat on the ground, as your fully extended body will provide a larger surface to conduct electricity. Stay in a tuck position well after the storm passes.
- Watch for local flooding you may have to move if water begins to accumulate.
- If you feel your hair stand on end in a storm, drop into the tuck position **immediately.** This sensation means electric charges are already rushing up your body from the ground toward an electrically charged cloud. Minimize your contact with the ground to minimize your injury.

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5. PROTECTIVE MEASURES

Personal Protective Equipment Requirements:

Copy and paste a checkmark "✓"into appropriate boxes.

Required PPE	Task 1	Task 2	Task 3	Task 4
	Test Pits	MW Install	Well Development	Well Sampling
Hard hat	✓	✓		
Safety glasses w/side shields	✓	✓	✓	✓
Steel-toe footwear	✓	✓	✓	✓
Hearing protection (plugs, muffs)	✓	✓		
Tyvek ™ coveralls				
PE-coated Tyvek™ coveralls				
Boots, chemical resistant				
Boot covers, disposable				
Leather work gloves				
Inner gloves - Nitrile	✓	✓	✓	✓
Outer gloves -				
Tape all wrist/ankle interfaces				
Half-face respirator*				
Full-face respirator*				
Organic vapor cartridges				
Acid gas cartridges				
Other cartridges: Enter type here				
P-100 (HEPA) filters				
Face shield				
Personal Flotation Device (PFD)				
High-Visibility Safety Vest	✓	✓	✓	✓
Other:				
Level of protection required [C or D]:	D	D	D	D

* In the event of respirator use, H&A staff must be medically qualified, fit tested and clean shaven with no facial hair that will interfere with the seal.

The required PPE checked in any box above must be on site during the task being performed. Work shall not commence unless the required PPE is present.

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Site Safety Equipment Requirements:

Check all items that are required to be on site.

Site Safety Equipment

☑ Fire Extinguisher	☑ First Aid Kit	☐ Flashlight
☐ Air horn/signaling device	☑ Cellular Phone	☐ Duct tape
☐ Ladder	☑ Barricade tape	☐ Drum dolly
☐ Two-way radio	☑ Safety cones	☐ Harness/Lanyard
☐ Other Specify		

The required equipment checked in any box above must be on site during the task being performed. Work shall not commence unless the equipment is present.

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6. MONITORING PLAN AND EQUIPMENT

Is air/exposure monitoring required at this work site for personal protection? ☑ Y ☐ N

Is perimeter monitoring required for community protection? ☐ Y ☑ N

Monitoring/Screening Equipment **Requirements:**

Check all items that are required to be on site.

Required Monitoring/Screening Equipment

- ☐ Photo-Ionization Detector (PID) 10.2eV
- ☐ Photo-Ionization Detector (PID) 11.7eV
- ☑ Photovac Micro Tip (PID) 10.6eV
- ☐ Organic Vapor Monitor (FID)
- ☐ Photovac Gas Chromatagraph (GC)
- ☐ Combustible Gas Indicator (CGI) (LEL)
- ☐ Multiple Gas Detector LEL/O2/H2S/CO
- ☐ Dust Monitors (RAMs)
- ☐ Colorimetric tubes
- ☐ Other

The required equipment checked in any box above must be on site. Work shall not commence unless the equipment is present.

Standard Action Levels and Required Responses:

Exposure Guidelines for common contaminants are listed in Table 1 - Occupational Exposure Limits in the Chemical Hazards section above.

Requirements for PPE upgrades based on monitoring are in Table 2 - Monitoring Methods, Action Levels and Protective Measures following the Specific Monitoring Requirements section below.

Action levels for readings obtained with a multiple gas detector are listed below.

Instrument	Normal	Operating levels	Action levels – required responses
Oxygen Meter	20.9%	Between 19.5-23.5%	Below 19.5 %: leave area, requires supplied air Above 23.5%: leave area, fire hazard
CGI	0%	Less than 10%	Greater than 10%: fire/explosion hazard; cease work
Hydrogen Sulfide	0%	Less than 10 ppm.	Greater than 15 ppm (or 10 ppm for 8 hrs) requires supplied air respirator
Carbon Monoxide	0%	Less than 25 ppm	Greater than 200 ppm for 1 hour (or 25 ppm for 8 hrs) requires supplied air respirator

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Standard Air Monitoring Plan (Volatiles):

- Prior to the beginning of work obtain background readings with the PID away from the site.
- Monitor the breathing zone when site soil is exposed (e.g., while drilling or excavating is occurring, etc.) with the PID.
- Monitoring should be conducted most frequently (e.g., every 15-30 minutes) when drilling or excavation first begins in a particular area and when soil is removed from the hole. After this, and if no exceedances of exposure limits are noted (see below), monitoring may be conducted less frequently (e.g., every 60 minutes).
- H&A general exposure limits will be used when a mixture of potentially volatile chemicals are suspected to be present in soil at the site.

In summary, if a reading of 10 ppm above background is detected with the PID for 5 minutes or longer, back away for a few minutes. Screen the air again after any vapors/gases have been given a chance to dissipate. If 10 ppm above background is still noted, evacuate the area and call the RHSC and PM for further guidance.

- Record monitoring data and PPE upgrades in field book or on Record of Field Monitoring form and maintain with project files.

- Air monitoring for exposure should be based on the frequency established under the Standard Air Monitoring Plan or under the Specific Monitoring Requirements. Record time, location and results of monitoring and actions taken based upon the readings.

Standard Dust Control Measures and Monitoring Plan:

Dust Control Measures:

It is anticipated that exposure to airborne dust can be mitigated during work operations as necessary to control dust emissions by means of limiting the area of exposed soils and through the use of water sprays. If dust emissions cannot be controlled by these standard measures, additional measures may be employed such as the use of a tackifier (if approved) to stabilize soil exposures or by covering exposed soil and stockpiles with tarpaulins, plastic sheeting or geotextile fabric. Otherwise cease work immediately and contact the Project Manager or the Corporate Health & Safety Manager for assistance. It is not permissible for dust emissions to escape from the site at any time and perimeter dust monitoring may be required to insure public safety.

Dust Monitoring:

Respirable Aerosol Monitors (RAM) can be used to monitor total dust levels in work zones and/or at the site perimeter. These instruments do not give specific readings of contaminant concentration (e.g. metals, asbestos, etc.). Depending upon the contaminants present, it may be mandatory for all workers to upgrade to level C protection using a half-face air-purifying respirator with HEPA (P-100) filters if dust levels cannot be adequately controlled during any of the on-site tasks. The H&A Site Safety Officer (SSO) will determine PPE upgrades based upon visual determination as necessary and the OSHA PEL for each known or suspected contaminant. The OSHA PEL/STEL for Respirable Nuisance Dust is 5 mg/m^3 (8 hour TWA).

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Action levels for fugitive dust at the site perimeter are based upon the daily PM_{10} dust standard of 0.15 mg/m^3 in the National Ambient Air Quality Standard for Inhalable Dust (NAAQS).

Personal dust monitoring using an industrial hygiene pump and a filter cassette may be conducted on each day of operations. In such cases samples are collected from workers with the greatest potential dust exposure and analyzed by an accredited laboratory for specific contaminants.

Specific Monitoring Requirements:

Monitoring requirements and frequency is indicated by task and location below.

Task Number:	1	Frequency	1	times per	15 min

Task Number:	2	Frequency	1	times per	15 min

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TABLE 2
Last Revised September 2002

MONITORING METHOD, ACTION LEVELS AND PROTECTIVE MEASURES

INSTRUMENT	HAZARD	ACTION LEVEL	ACTION RESPONSE
Respirable Dust Monitor	Total Particulates	> 5 mg/m^3	Upgrade to Level C Protection
OVA, HNU[2], Photovac Microtip	Total Organic Vapors	Background 10 ppm > background or lowest OSHA permissible exposure limit, whichever is lower, or as modified for this task. Sustained for >5 minutes in the breathing zone. 50 ppm over background, unless lower values required due to respirator protection factors	Level D Protection Upgrade to Level C - site evacuation may be necessary for specific compounds Cease work; upgrade to Level B[3] may be required
Explosimeter[4] (LEL)	Flammable/Explosive Atmosphere	<10% Scale Reading 10-15% Scale Reading >15% Scale Reading	Proceed with work Monitor with extreme caution Evacuate site
0xygen Meter[5]	Oxygen-Deficient Atmosphere	19.5% - 23.5% 0_2 < 19.5% 0_2 > 23.5% 0_2	Normal - Continue work Evacuate site; oxygen deficient Evacuate site; fire hazard
Radiation Meter[6]	Ionizing Radiation	0.1 Millirem/Hour > 1 Millirem/Hour	If > 0.1, radiation sources may be present[7] Evacuate site; radiation hazard
Drager Tubes	Vapors/Gases	Species Dependent > 1 ppm vinyl chloride > 1 ppm benzene > 1 ppm 1,1-DCE	Consult Table 1 or other resources for concentration toxicity/detection data. Upgrade to Level C if concentration of compounds exceed thresholds shown at left; May need to cease work if other levels exceeded - site specific
Gas Chromatograph (GC)	Organic Vapors	3 ppm total OV > background or > lowest specific OSHA permissible exposure limit, whichever is lower	On-site monitoring or tedlar bag sample collection for off-site/laboratory analysis

Notes:
1. Monitor breathing zone.
2. Can also be used to monitor some inorganic species.
3. Positive pressure demand self contained breathing apparatus
4. Lower explosive limit (LEL) scale is 0-100%. LEL for most gasses is 15%.
5. Normal atmospheric oxygen concentration at sea level is 20%
6. Background gamma radiation is ~0.01-0.02 millirems/hour.
7. Contact H&A Health and Safety staff immediately.

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Calibration and Use of Equipment:

Calibrate all monitoring equipment in accordance with manufacturers requirements, H&A calibration (OP) standards and site specific requirements (e.g., at the beginning and end of each work day). Calibration of equipment shall be documented in the field notes or Daily Field Report (DFR). Documentation should include:

- Date/time
- Zero reading before calibration
- Concentration of calibration gas
- Reading obtained with calibration gas before adjusting span\
- Final reading obtained with calibration gas after adjusting span

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7. DECONTAMINATION AND DISPOSAL METHODS

Personal Hygiene Safeguards:

The following minimum personal hygiene safeguards shall be adhered to:

- No smoking or tobacco products on any Hazwoper project.
- No eating or drinking in the exclusion zone.
- It is required that personnel present on site wash hands before eating, smoking, taking medication, chewing gum/tobacco, using the restroom, or applying cosmetics and before leaving the site for the day.
- It is recommended that personnel present on site shower or bathe at home at the end of each day of working on the site.

Standard Personal Decontamination Procedures:

Outer gloves and boots should be decontaminated periodically as necessary and at the end of the day. Brush off solids with a hard brush and clean with soap and water or other appropriate cleaner whenever possible. Remove inner gloves carefully by turning them inside out during removal. Wash hands and forearms frequently. It is good practice to wear work-designated clothing while on-site which can be removed as soon as possible. Non-disposable overalls and outer work clothing should be bagged onsite prior to laundering. If gross contamination is encountered on-site contact the Project Manager and RHSC to discuss proper decontamination procedures. The steps required for decontamination will depend upon the degree and type of contamination but will generally follow the sequence below.

1. Remove and wipe clean hard hat
2. Rinse boots and gloves of gross contamination
3. Scrub boots and gloves clean
4. Rinse boots and gloves
5. Remove outer boots
6. Remove outer gloves
7. Remove Tyvek coverall
8. Remove respirator, wipe clean and store
9. Remove inner gloves

Location of Decontamination Station:

N/A

Disposal of PPE:

PPE that is not grossly contaminated can be bagged and disposed in regular trash receptacles. PPE that is grossly contaminated must be bagged (sealed) and field personnel should communicate with the Project Manager to determine proper disposal.

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Tools & Equipment Decontamination:

All decontamination should be conducted at the site and not at the office or lab.

Check all equipment and materials needed for decontamination of tools and other equipment.

☐ Acetone	☑ Distilled water	☑ Poly sheeting
☑ Alconox soap	☑ Drums for water	☐ Steam cleaner
☑ Brushes	☐ Hexane	☐ Tap water
☑ Disposal bags	☐ Methanol	☑ Washtubs
☑ 5 gallon pails	☑ Other Paper towels	

Standard Equipment Decontamination Procedures:

Air monitoring instrumentation and delicate instruments that are difficult to decontaminate or sensitive to water should be protected from contamination during use through the use of plastic sheeting. To the extent possible, efforts should be taken to limit the degree of contamination to hand tools and sampling equipment during use. Proper PPE must be worn while performing decontamination, including the wearing of chemical safety goggles and gloves. Storage or transport of decontamination solvents in squirt bottles is not permitted as they may discharge their contents upon ambient temperature change or leak if overturned. Standard equipment decontamination procedures are as follows. Any additional requirements are listed under Specific Equipment Decontamination Procedures below.

Pretreatment of heavily contaminated equipment may be conducted as necessary:

1. Remove gross contamination using a brush or wiping with a paper towel
2. Soak in a solution of Alconox and water (if possible)
3. Wipe off excess contamination with a paper towel
4. Clean with hexane or acetone and allow to dry

Standard decontamination procedure:

1. Wash using a solution of Alconox and water
2. Rinse with potable water
3. Rinse with methanol
4. Rinse with distilled water

Specific Equipment Decontamination Procedures:

Not Applicable

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Standard Disposal Methods for Contaminated Materials:

Excess sample solids, decontamination materials, rags, brushes, poly sheeting, etc. that are determined to be free of contamination through field screening can usually be disposed into client-approved, on-site trash receptacles. Uncontaminated wash water may be discarded onto the ground surface away from surface water bodies in areas where infiltration can occur. Contaminated materials must be segregated into liquids or solids and drummed separately for off site disposal. Any additional requirements are listed under Specific Disposal Methods for Contaminated Materials below.

Specific Disposal Methods for Contaminated Materials:

Not Applicable

Disposal Methods for Contaminated Soils:

Contaminated soil cuttings and spoils must be drummed for disposal off-site unless otherwise specifically directed. Soil cuttings and spoils determined to be free of contamination through field screening can usually be returned to the boreholes or excavations from which they came. Any additional requirements are listed under Specific Disposal Methods for Contaminated Soils below.

Specific Disposal Methods for Contaminated Soils:

Not Applicable

Contaminated Soil Sent to Geotechnical Lab:

Assignments that include geotechnical lab testing on contaminated samples must be accompanied with written data that will provide information on the type and extent of contamination. Project Managers must communicate any anticipated or known chemical hazards to the lab when assigning geotechnical tests. Preferably, a copy of this HASP should be forwarded to the laboratory for their review. If the contamination is not known, the PM must contact the laboratory and discuss the source of the sample to help identify any potential hazards that may be associated with the sample.

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8. CONTINGENCY PLANNING

How H&A responds to an emergency depends on whether we are at an active facility or another other location. Many active facilities have very stringent requirements for the mitigation of emergencies. Therefore, the PM is responsible for identifying any specific requirements from the client contact.

As a rule of thumb, the following are H&A's basic responses to handling Emergencies. Typically, H&A does not mitigate emergencies. When Clients request or require specific functions such as First Aid/CPR trained personnel on site, we typically conform. Before any Project Manager or RHSC agrees to something more stringent, many issues should be considered such as training, safety, feasibility of an adequate response, insurance requirements, and much more.

Fire:

- Major Fires - Major fires will be mitigated by the local fire departments or by client's on-site fire/emergency response departments.
- Incipient Stage Fires -Incipient stage fires will be extinguished by on-site personnel using fire extinguishers. Only those who have received annual training may use an extinguisher.

Medical:

All H&A employee injuries and illnesses will be documented using the Supervisor's Accident / Injury / Near Miss Report (SAIR). This form is available on the Intranet.

- First Aid - First aid will be addressed using the on-site first aid kit. H&A employees are not required or expected to administer first aid/CPR to any H&A, Contractor, or Civilian personnel at any time and it is H&A's position that those who do are doing it on their behalf and not as a function of their job.
- Trauma - Based upon the nature of the injury, the injured party may be transported to the nearest hospital or emergency clinic by on-site personnel or by ambulance. First response to a trauma incident is to call 911 or facility security. H&A staff members are expected to assist in ancillary roles only such as directing ambulances to the scene. It is the discretion of the staff member on site whether an ambulance should be procured in remote locations where ambulance services will not be effective.

Hazardous Materials Spill:

- Small incidental spills (e.g. pint of motor oil) caused by H&A employees and/or by the contractor will be mitigated by the H&A staff member and/or the contractor.
- Large spills (e.g. large leak from heavy equipment fuel tank). The contractor is responsible for cleanup. In the event that it posses a serious human or environmental threat, the local Fire Department and/or client emergency response department will be contacted. Once emergency has been mitigated typically clean up will be provided by a vendor.

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Rescue:

H&A employees will not enter any confined spaces for rescue purposes.

Weather Related Emergencies:

H&A employees and their subcontractors should be aware of potential health effects and/or physical hazards of working during inclement weather. If applicable, safeguards against the effects and hazards of heat stress, cold stress, frostbite, thunderstorms, and lightning, etc., should be included with the section pertaining to physical hazards in this HASP.

Evacuation Alarms:

Evacuation alarms and/or emergency information will be communicated among personnel on site through verbal communication.

Emergency Services:

Emergency services will be summoned via on-site or cellular phone.

Emergency Evacuation Plan:

The site evacuation plan is as follows:

1. Establish a designated meeting area to conduct a head count in the event of an emergency evacuation.
2. If the work area is not near an emergency exit, exit via the closest route and meet at the designated meeting area.
3. Notify emergency response personnel (fire, police and ambulance) of the number of missing or unaccounted for employees and their suspected location.
4. Administer first aid will in the meeting area as necessary.

Under no circumstances should any personnel re-enter the site area without the approval of the corporate H&S manager, the H&S coordinator, and the fire department official in charge.

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9. HEALTH & SAFETY PLAN ACKNOWEDGMENT FORM

Note: Only H&A employees sign this page.

I hereby acknowledge receipt and briefing on this Health & Safety Plan prior to the start of on-site work and declare that I understand and agree to follow the provisions and procedures set forth herein while working on this site.

PRINTED NAME	SIGNATURE	DATE

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10. PRE-JOB SAFETY CHECKLIST

The following checklist is designed to help Project Managers verify that all Health & Safety requirements are satisfied for projects involving site work and to aid in the preparation of the site-specific HASP.

Please initial and date the appropriate box once each requirement has been satisfied prior to commencement of site work.

#	Project H&S Requirements	Approval by PM or RHSC (initial each box or place NA)	Date Approved
1	Project site history has been researched and summarized, current site conditions have been determined and documentation of previous investigations, risk analyses and chemical data has been assembled and summarized.		
2	Project work scope has been outlined and potential chemical and physical hazards associated with work tasks have been identified.		
3	Task Safety Analysis has been performed and attached to the HASP.		
4	H&A personnel to be involved with the project have been identified and are current with medical surveillance, OSHA 40 hour and 8 hour refresher training. Hazwoper site supervisor requirements are satisfied.		
5	Additional training requirements have been met: e.g. nuclear density gauge, DOT, Confined Space Entry, Competent Person Training for Excavation, OSHA 10 hour certification, Railway Safety Training, etc.		
6	H&A personnel that may be required to wear a respirator are medically qualified and have current certification of fit testing.		
7	Client's additional H&S requirements have been met: e.g. facility safety orientations, safety documentation, meetings, special PPE requirements		
8	H&A subcontractors have met H&A's minimum requirements including: current OSHA 40 hour training, medical surveillance, written HASP, insurance, MSDSs.		
9	MSDSs are on site and available for chemicals on site.		
10	Safety equipment is available: e.g. flashlight, telephone, ladders, traffic cones, barricade tape, fire extinguisher, first aid kit, PPE, respiratory protection, air and dust monitoring instrumentation (calibrated), personal flotation device (PFD), 90' life line with ring, decontamination equipment, etc.		
11	HASP and supporting documentation is complete and signed by all members.		

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APPENDIX A
HASP Amendment Form

This Appendix is to be used whenever there is an immediate change in the project scope that would require an amendment to the HASP. For project scope changes associated with "add-on" tasks, the changes must be made in the body of the HASP. Before changes can be made, a review of the potential hazards must be initiated by the H&A Project Manager.

Amendment No.	
Site Name:	
Work Assignment No.:	
Date:	
Type of Amendment:	
Reason for Amendment:	
Alternate Safeguard Procedures:	
Required Changes in PPE:	

Project Manager Signature: _____ Date: _____

Regional Health and Safety Coordinator: _____ Date: _____

This original form must remain on site with the original HASP. If additional HASPs are in the field, it is the Project Manager's responsibility to forward a signed copy of this amendment to those who have copies.

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APPENDIX B
Issuance and Compliance
Site Safety Officer Role and Responsibilities
Training Requirements

This Health & Safety Plan (HASP) has been prepared in accordance with the requirements of Title 29 the Code of Federal Regulations (CFR) Section 1910.120/1926.65 to provide guidance for the protection of onsite personnel from physical harm and chemical exposure while working at the subject site.

The specific requirements of this HASP include precautions for hazards that exist during this project and may be revised as new information is received or as site conditions change.

- This HASP must be signed by all Haley & Aldrich (H&A) staff members who will work on the project, including H&A visitors. By signing the Health and Safety Plan Acknowledgement Form personnel are acknowledging that they are aware of the specific hazards of the site and agree to follow the provisions and procedures required to safeguard themselves and others from those hazards.
- This HASP or a current signed copy must be retained at the site at all times when H&A staff members are present.
- Deviations from this HASP are not permitted without prior approval from the above signed. Unauthorized deviations may constitute a violation of H&A company procedures/policies and may result in disciplinary action.
- Revisions to this HASP must be outlined within the contents of the HASP. If immediate or minor changes are necessary, the RHSC and H&A Project Manager may use Appendix A (HASP Amendment Form), located in the back of this HASP. Any revision to the HASP requires personnel to be informed of the changes and that they understand the requirements of the change.
- This HASP is not for H&A Subcontractor use. Each subcontractor engaged is responsible for all matters relating to the health and safety of their personnel and the safe operation of their equipment. This HASP will be made available as a reference so that subcontractors are informed of the potential hazards associated with the site to the extent we are aware. Subcontractors must develop their own HASP which must be, at a minimum, at least as protective as this HASP.
- This Site Specific HASP provides only site-specific descriptions and work procedures. General safety and health compliance programs in support of this HASP (e.g., injury reporting, medical surveillance, personal protective equipment (PPE) selection, etc. are described in detail in the H&A Corporate Health and Safety Program Manual and within Standard Operating Procedures (OPs). Both the manual and OPs can be located on the Company Intranet. When appropriate, users of this HASP should always refer to these resources and incorporate to the extent possible. The manual and OPs are available to clients and regulators per request.

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Site Safety Officer:

The site safety officer (SSO) is defined as the individual responsible to the employer with the authority and knowledge necessary to implement the HASP and verify compliance with applicable health and safety requirements.

The H&A Project Manager may designate any person as the site safety officer (SSO) and determines the order of authority on site. Usually the highest ranking person on site is the SSO. A site safety officer must be on site at all times. When none of the designated SSOs are present on site, the senior person for H&A on site will default to the SSO. This project has identified the following hierarchy for SSO.

1. Enter name of site safety officer here

2. Enter name of site safety officer here

Site Safety Officer Roles and Responsibilities:

The SSO is responsible for field implementation of this HASP and enforcement of safety rules and regulations. SSO functions include:

- Act as H&A's liaison for health and safety issues with client, staff, subcontractors, and agencies.
- Verify that utility clearance has been performed by H&A subcontractors.
- Oversee day-to-day implementation of the HASP by H&A employees on site.
- Interact with subcontractor project personnel on health and safety matters.
- Verify use of required PPE as outlined in the HASP.
- Inspect and maintain H&A safety equipment, including calibration of air monitoring instrumentation used by H&A.
- Perform changes to HASP and document in Appendix A of the HASP as needed and notify appropriate persons of changes.
- Investigate and report on-site accidents and incidents involving H&A and its subcontractors.
- Verify that site personnel are familiar with site safety requirements (e.g., the hospital route and emergency contact numbers).
- Report accidents, injuries, and near misses to the H&A PM and Regional Health and Safety Coordinator (RHSC) as needed.

The SSO will conduct initial site safety orientations with site personnel (including subcontractors) and conduct toolbox and safety meetings thereafter with H&A employees and H&A subcontractors at regular intervals and in accordance with H&A policy and contractual obligations. The SSO will track the attendance of site personnel at H&A orientations, toolbox talks, and safety meetings. Subcontractors will document training and provide training rosters to the H&A SSO.

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The SSO will report accidents such as injury, overexposure, or property damage to the Regional Health and Safety Coordinator, to the Project Manager, and to the safety managers of other on-site consultants and contractors. The SSO will consult with the safety managers of other on-site consultants and subcontractors on specific health and safety issues arising over the course of the project, as needed.

Health and Safety Training Requirements:

Personnel will not be permitted to supervise or participate in field activities until they have been trained to a level required by their job function and responsibility. H&A staff members, contractors, subcontractors, and consultants who have the potential to be exposed to contaminated materials or physical hazards must complete the training described in the following sections.

The H&A Project Manager/RHSC will be responsible for maintaining and providing to the client/site manager documentation of H&A staff members' compliance with required training as requested. Records shall be maintained per OSHA requirements.

40-Hour Health and Safety Training

The 40-Hour Health and Safety Training course provides instruction on the nature of hazardous waste work, protective measures, proper use of personal protective equipment, recognition of signs and symptoms which might indicate exposure to hazardous substances, and decontamination procedures. It is required for all personnel working on-site, such as equipment operators, general laborers, and supervisors, who may be potentially exposed to hazardous substances, health hazards, or safety hazards consistent with 29 CFR 1910.120.

8-hour Annual Refresher Training

Personnel who complete the 40-hour health and safety training are subsequently required to attend an annual 8-hour refresher course to remain current in their training. When required, site personnel must be able to show proof of completion (i.e., certification) at an 8-hr refresher training course within the past 12 months.

8-Hour Supervisor Training

On-site managers and supervisors directly responsible for, or who supervise staff members engaged in hazardous waste operations, should have eight additional hours of Supervisor training in accordance with 29 CFR 1910.120. Supervisor Training includes, but is not limited to, accident reporting/investigation, regulatory compliance, work practice observations, auditing, and emergency response procedures.

Additional Training for Specific Projects

H&A personnel will ensure their personnel have received additional training on specific instrumentation, equipment, confined space entry, construction hazards, etc., as

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necessary to perform their duties. This specialized training will be provided to personnel before engaging in the specific work activities including:

- Client specific training or orientation
- Competent person excavations
- Confined space entry (entrant, supervisor, and attendant)
- Heavy equipment including aerial lifts and forklifts
- First aid/ CPR
- Diving certification
- Use of fall protection
- Commercial drivers license
- Use of nuclear density gauges
- Asbestos awareness

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