

2008 GROUNDWATER INVESTIGATION WORK PLAN

**GENERAL MOTORS CORPORATION
GRAND RAPIDS METAL PLANT
WYOMING, MICHIGAN**

**AUGUST 2008
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TABLE OF CONTENTS

	<u>Page</u>
1.0 INTRODUCTION.....	1
1.1 SITE BACKGROUND	1
1.2 PURPOSE OF 2008 GROUNDWATER INVESTIGATION WORK PLAN	1
2.0 CURRENT CONDITIONS	2
2.1 NATURE AND EXTENT OF CONTAMINATION	2
3.0 SCOPE OF WORK.....	3
3.1 SOIL BORING AND MONITORING WELL INSTALLATION.....	3
3.1.1 SOIL BORING INSTALLATION/VERTICAL AQUIFER SAMPLING.....	3
3.1.2 MONITORING WELL INSTALLATION	4
3.1.3 MONITORING WELL RE-INSTALLATION	5
3.1.4 MONITORING WELL DEVELOPMENT.....	6
3.1.4 SURVEYING.....	6
3.1.6 DECONTAMINATION	6
4.0 SAMPLING PROCEDURES	8
4.1 SOIL SAMPLE COLLECTION.....	8
4.2 DISCRETE GROUNDWATER SAMPLE COLLECTION	8
4.3 GROUNDWATER SAMPLE COLLECTION.....	9
4.4 LNAPL SAMPLING.....	9
4.5 DECONTAMINATION	9
4.6 WASTE CHARACTERIZATION	10
5.0 ANALYTICAL PROTOCOLS.....	11
6.0 REPORTING	12
7.0 SCHEDULE	13

LIST OF FIGURES
(Following Text)

FIGURE 1.1	SITE LOCATION
FIGURE 1.2	SITE PLAN
FIGURE 3.1	PROPOSED HYDROPUNCH AND MONITORING WELL LOCATIONS

LIST OF TABLES
(Following Text)

TABLE 3.1	SAMPLING AND ANALYSIS PLAN

LIST OF APPENDICES
(Following Text)

APPENDIX A	QUALITY ASSURANCE PROJECT PLAN

LIST OF ACRONYMS

COC	Chain of Custody
CRA	Conestoga-Rovers & Associates
DO	Dissolved Oxygen
DOT	Department of Transportation
GM	General Motors Corporation
HSA	Hollow Stem Auger
MDEQ	Michigan Department of Environmental Quality
ORP	Oxidation-Reduction Potential
PID	Photoionization Detector
PVC	Polyvinyl Chloride
QAPP	Quality Assurance Project Plan
QA/QC	Quality Assurance/Quality Control
RCI	Reactivity, Corrosively, and Ignitability
SOW	Scope of Work
SVE	Soil Vapor Extraction
SVOCs	Semi-Volatile Organic Compounds
TAT	Turn-Around-Time
TCLP	Toxicity Characteristic Leaching Procedure
TCL	Target Compound List
TOC	Total Organic Carbon
VAS	Vertical Aquifer Sampling
VOCs	Volatile Organic Compounds
WWTP	Wastewater Treatment Plant

1.0 INTRODUCTION

The purpose of this 2008 Groundwater Investigation Work Plan (Work Plan) is to present the protocol to be used for the implementation of the installation of a soil boring and two monitoring wells, and the collection and analysis of groundwater samples at the General Motors Corporation (GM) Grand Rapids Metal Plant located at 300 36th Street in Wyoming, Michigan (Site). Figure 1.1 presents the Site location.

1.1 SITE BACKGROUND

The Site background is presented in the Work Plan and the Data Report submitted to the Michigan Department of Environmental Quality (MDEQ) under separate cover on November 24, 2002 and March 2, 2004, respectively. A Site plan is presented on Figure 1.2.

1.2 PURPOSE OF 2008 GROUNDWATER INVESTIGATION WORK PLAN

The purpose of this Work Plan is to outline the activities proposed to further evaluate and delineate chlorinated compound and semi-volatile organic compound (SVOC) impacts to groundwater above the Michigan Act 451, Part 201 Residential Cleanup Criteria, and to further define hydrogeologic conditions at the Site.

2.0 CURRENT CONDITIONS

2.1 NATURE AND EXTENT OF CONTAMINATION

The nature and extent of contamination is presented in the 2005-2006 Data Report submitted to the MDEQ under separate cover on April 30, 2007. Additionally, semi-annual sampling is conducted at the Site and the results are provided in individual reports submitted to the MDEQ under separate cover on a semi-annual basis. Semi-annual groundwater sampling for 2008 will be conducted consistent with the scope of work presented in the 2005 Groundwater Monitoring Work Plan submitted to the MDEQ under separate cover on April 12, 2005. Target Compound List (TCL) volatile organic compounds (VOCs) and TCL semi-volatile organic compounds (SVOCs) were selected for investigation and reporting because they were detected in previous rounds of groundwater sampling and remain on the list as the chemicals of concern.

2.2 SITE GEOLOGY AND HYDROGEOLOGY

Information regarding the geologic conditions at the Site was collected during previous environmental investigations. Site geology and hydrogeology are presented in the 2005-2006 Data Report submitted to the MDEQ under separate cover on April 30, 2007.

3.0 SCOPE OF WORK

The SOW for this Work Plan includes the advancement of one soil boring and the installation of two monitoring wells, and the collection and analysis of approximately eight groundwater samples, including QA/QC samples. Additionally, the SOW includes the analysis of select groundwater samples, collected during the October 2008 Semi-Annual Groundwater Monitoring event, for SVOCs, in addition to those locations previously identified in the 2005 Groundwater Monitoring Work Plan. The investigation field work will adhere to the methods and procedures specified in this Work Plan, including the Quality Assurance Project Plan (QAPP) presented in Appendix A.

3.1 SOIL BORING AND MONITORING WELL INSTALLATION

The proposed soil boring and monitoring well locations to further evaluate and delineate chlorinated compound and SVOC impacts to groundwater above the Michigan Act 451, Part 201 Residential Cleanup Criteria and to further define hydrogeologic conditions at the Site are presented on Figure 3.1. The soil boring and monitoring wells will be installed as outlined below.

3.1.1 SOIL BORING INSTALLATION/VERTICAL AQUIFER SAMPLING

One soil boring, HP18-08, will be advanced on the south side of 35th street between Hillcroft Avenue and Birchwood Avenue to evaluate potential chlorinated compound contamination in groundwater.

The soil boring will be advanced at the designated location using a rotary drill rig equipped with a 4 ¼-inch inside diameter hollow stem auger (HSA).

Soil samples will be collected continuously using 2-foot long, 2-inch outside diameter split spoon samplers at HP18-08 during installation of the soil boring, as presented in Section 4.1. Soil samples will be collected from the borings in accordance with the Standard Penetration Test Method, ASTM D1586 and as presented in Section 4.1. The split spoon samples will be examined by a CRA geologist for visual/olfactory evidence of impact and screened with an 11.7 eV bulb photoionization detector (PID). If visual/olfactory evidence of impact or high PID readings are observed, at the discretion of CRA field personnel, soil samples will be collected and submitted to the laboratory for chemical analysis. Additionally, discrete groundwater samples will be collected

from the soil boring utilizing vertical aquifer sampling (VAS) via a hydropunch, as presented in Section 4.2.

Soil cuttings will be screened with an 11.7 eV bulb PID and examined for visual/olfactory indication of contamination. All soil cuttings will be containerized in Department of Transportation (DOT) approved 55-gallon drums and labeled for future characterization.

Upon completion of discrete groundwater sample collection, the borehole will be abandoned using the tremie-method to backfill the borehole annulus with a high solids bentonite grout to ground surface.

3.1.2 MONITORING WELL INSTALLATION

One monitoring well, MW18-08, will be installed to evaluate potential chlorinated compound contamination in groundwater.

The monitoring well will be installed utilizing a 4 ¼-inch HSA. MW18-08 will be installed adjacent to HP18-08. Split spoon samples will not be collected from MW18-08 during monitoring well installation as the monitoring well will be installed adjacent to HP18-08 and it can be assumed that the stratigraphy will be the same as that of the HP18-08. A split spoon sample will be collected upon reaching depth during the installation of monitoring wells to confirm that the geology is the same.

Soil cuttings will be screened with an 11.7 eV bulb PID and examined for visual/olfactory indication of contamination. All soil cuttings will be containerized in DOT approved 55-gallon drums and labeled for future characterization.

Monitoring well MW18-08 will be constructed using Schedule 40 polyvinyl chloride (PVC) risers and 5-foot long, 0.01-inch slot Schedule 40 PVC screen through the augers as a 2-inch diameter installation. During completion of the installation, the riser pipe shall be temporarily capped to prevent entrance of foreign materials during overburden monitoring well installation. The screen and casing will be joined utilizing flush threaded joints. MW18-08 will be installed at a depth consistent with the maximum detected levels of VOCs during the VAS. If there are no VOCs detected during the VAS, the screen will be set at the same elevation as the screen for MW4-03.

An inert silica sandpack will be placed to a height of three vertical feet above the top of the screens using a flush-threaded 1-inch diameter tremie pipe as the augers are

withdrawn from the borehole. If bridging of the silica sand occurs, the bridged material will be mechanically broken prior to the addition of more silica sand.

The remainder of the annulus will be filled with a high solids bentonite grout using a flush-threaded 1-inch diameter tremie pipe to within two feet of the ground surface. The grout will be set in one continuous operation with the entire amount placed before the initial set occurs. Once the grout has set, the remaining portion of the unfilled annulus will be filled with concrete and a flushmount protective surface casing will be installed at the well. The protective flushmounted casing will be centered over the riser and extend into the concrete collar 1-foot. The riser pipe will be capped with an expandable locking cap. Locks will be installed with one set of keys kept at the Site and one set kept by CRA.

The monitoring well will be clearly labeled with a unique identification number. The number will be located on the well protector in an area where the number is protected from possible tampering.

The stratigraphic boring log from the newly installed monitoring well will be used in conjunction with the stratigraphic boring logs from previous drilling activities to supplement previous data to further determine geologic and hydrogeologic conditions in the area of the chlorinated compound impact.

3.1.3 MONITORING WELL RE-INSTALLATION

Based on observed groundwater levels, during portions of the year, groundwater levels have been identified to be above the top of the current screened interval of monitoring well 85-3 (20.3 to 30.3 feet bgs). As such, the LNAPL monitoring currently conducted in this well is not representative of the potential LNAPL associated with the water table in this portion of the plant.

Monitoring well 85-3 will be overdrilled utilizing a 4 ¼-inch HSA, with the existing monitoring well removed, and a new monitoring well, MW19-08, installed in the same location to monitor the presence of LNAPL. The new monitoring well will be screened to straddle the water table, from 15 to 25 feet bgs, in order to monitor the LNAPL historically observed in this portion of the plant. The new monitoring well will be installed consistent with the protocols described in Section 3.1.2, with the exception that MW19-08 will be constructed using Schedule 40 PVC risers and 10-foot long, 0.01-inch slot Schedule 40 PVC screen through the augers as a 2-inch diameter installation.

Upon completion of monitoring well MW19-08, a passive skimmer will be installed in the monitoring well for collection of a LNAPL sample for "fingerprinting" purposes, if sufficient volume of LNAPL is present for analysis.

3.1.4 MONITORING WELL DEVELOPMENT

Groundwater level measurement and corresponding bottom depths will be recorded for MW18-08 and MW19-08. The well volumes will be calculated and the wells will be developed by surging the wells with a stainless steel bailer for a minimum of five minutes and then purging with a submersible pump. The wells will be developed by removing a minimum of five well volumes until groundwater is developed to a silt-free condition, if possible, and the turbidity, pH, temperature, and conductivity of the groundwater have stabilized.

All generated development water will be visually examined and screened with an 11.7 eV bulb PID. Purge and development groundwater will be containerized in DOT approved 55-gallon drums and labeled for future characterization and disposal.

3.1.4 SURVEYING

A survey of the Site will be completed for the VAS location and the monitoring well locations. Soil boring and monitoring well locations and elevations will be surveyed, with elevations to the nearest 0.01-foot. These elevations will be referenced to a designated above mean sea level benchmark. The monitoring well elevations will be used to determine groundwater elevation, which will be used to develop Site groundwater contours, flow direction, and flow velocities.

3.1.6 DECONTAMINATION

Upon mobilization to the Site and prior to drilling commencement, the drill rig and all associated equipment will be thoroughly cleaned using a high pressure, low volume steam wash and inspected. Before initiating drilling at each subsequent location, the augers, drill rod, and other associated equipment will be decontaminated to prevent cross-contamination. Decontamination of sampling equipment is presented in Section 4.5.

All generated decontamination water will be visually examined and screened with a PID. All decontamination water will be containerized in DOT approved 55-gallon drums for future characterization and disposal.

4.0 SAMPLING PROCEDURES

The proposed sample locations are presented on Figure 3.1. All sampling will be performed in accordance with the procedures outlined below.

4.1 SOIL SAMPLE COLLECTION

Soil samples will be collected continuously utilizing 2-foot long, 2-inch diameter split spoon samplers from soil boring HP18-08. Split spoon samples will be examined by a CRA geologist for visual/olfactory evidence of impact and screened with an 11.7 eV bulb PID meter. If visual/olfactory evidence of impact or high PID meter readings are observed, at the discretion of CRA field personnel, soil samples will be collected and submitted to the laboratory for chemical analysis.

4.2 DISCRETE GROUNDWATER SAMPLE COLLECTION

VAS will be conducted at HP18-08 at 10-foot intervals beginning at the water table and continuing until the clay confining layer is encountered. Fifteen-foot intervals may be utilized at the discretion of the CRA field geologist, dependent upon conditions encountered in the field.

A 5-foot long, 2-inch diameter stainless steel hydropunch sampler with a 0.01-inch slotted screen and a well point will be driven through the undisturbed material through a 4 ¼-inch HSA. The well point will detach from the hydropunch sampler at the correct sample depth and groundwater will flow into, and fill the hydropunch sampler. The amount of time required to fill the hydropunch is dependent upon many factors including type of media (e.g., sand vs. clay) and depth below ground surface (i.e., increasing water pressure with increasing depth).

After sufficient time to fill the hydropunch sampler has passed, the sampler will be removed from the soil boring, the screen broken off, and the discrete groundwater sample will be collected from the sampler.

Approximately eight discrete groundwater samples, including QA/QC samples, will be collected and analyzed for TCL VOCs on a 48-hour turnaround time. Table 3.1 presents the Sampling and Analysis Plan.

Discrete groundwater samples will be placed into pre-cleaned laboratory provided containers, labeled, and submitted to the laboratory under chain-of-custody (COC) protocol.

4.3 GROUNDWATER SAMPLE COLLECTION

Groundwater samples will be collected and analyzed as directed by the 2005 Groundwater Monitoring Work Plan, which was submitted under separate cover on April 12, 2005.

In addition to the locations and parameters identified in the 2005 Groundwater Monitoring Work Plan, groundwater samples will be collected from monitoring wells 85-7, 87-8, and MW14-04 for analysis for TCL SVOCs during the October 2008 Semi-Annual Groundwater Monitoring event.

4.4 LNAPL SAMPLING

One LNAPL sample will be collected from the installed passive skimmer installed in newly installed monitoring well MW19-08 for analysis for TCL VOCs, TCL SVOCs, TAL metals, PCBs, density, and mean molecular weight. Table 3.1 presents the Sampling and Analysis Plan.

The LNAPL sample will be placed into pre-cleaned laboratory provided containers, labeled, and submitted to the laboratory under COC protocol.

4.5 DECONTAMINATION

All non-disposable sampling equipment will be decontaminated prior to each use by using an Alconox wash, potable water rinse, followed by a deionized water rinse and allowed to air dry. This includes split spoon samplers, the hydropunch, hand sampling tools, etc.

All generated decontamination water will be visually examined and screened with a PID. All decontamination water will be containerized in DOT approved 55-gallon drums for future waste characterization and disposal.

4.6 WASTE CHARACTERIZATION

All generated soil cuttings and decontamination/development water will be visually examined and screened with a PID. All generated soil cuttings and decontamination/development water will be containerized in DOT approved 55-gallon drums for future waste characterization and disposal.

Drum contents will be segregated, based on evidence of impact observed during field screening activities. One soil waste characterization sample will be collected from the drums and analyzed for Toxicity Characteristic Leaching Procedure (TCLP) metals, TCLP VOCs, TCLP SVOCs, reactivity, corrosivity, and ignitability (RCI), and total VOCs on a two-week turnaround time for off-Site disposal. Table 3.1 presents the Sampling and Analysis Plan.

Based on the analytical results from the VAS, decontamination/development water will be discharged to the on-Site wastewater treatment plant (WWTP) or properly disposed of off-Site.

5.0 ANALYTICAL PROTOCOLS

Groundwater samples will be analyzed for TCL VOCs and TCL SVOCs, as presented in the SOW and on Table 3.1. Additionally, the LNAPL sample will be analyzed for TCL VOCs, TCL SVOCs, TAL metals, PCBs, density, and mean molecular weight, as presented in the SOW and on Table 3.1. All samples will be analyzed using the United States Environmental Protection Agency approved methods set forth in "Test Methods for Evaluating Solid Waste, Physical/Chemical Methods", SW-846, 3rd Edition and Promulgated Update, November 1986, "Standards Methods for the Examination of Water and Wastewater", APHA, 19th Edition, 1995, and "Methods for Chemical Analysis of Water and Waste", EPA-600/4-79-020, revised March 1983. Quality Control/Quality Assurance samples will be collected for data and sample quality assessment in accordance with the QAPP. The QAPP is presented in Appendix A.

Following receipt of final analytical data, a data quality assessment and validation will be prepared by a CRA chemist, in accordance with the QAPP presented in Appendix A.

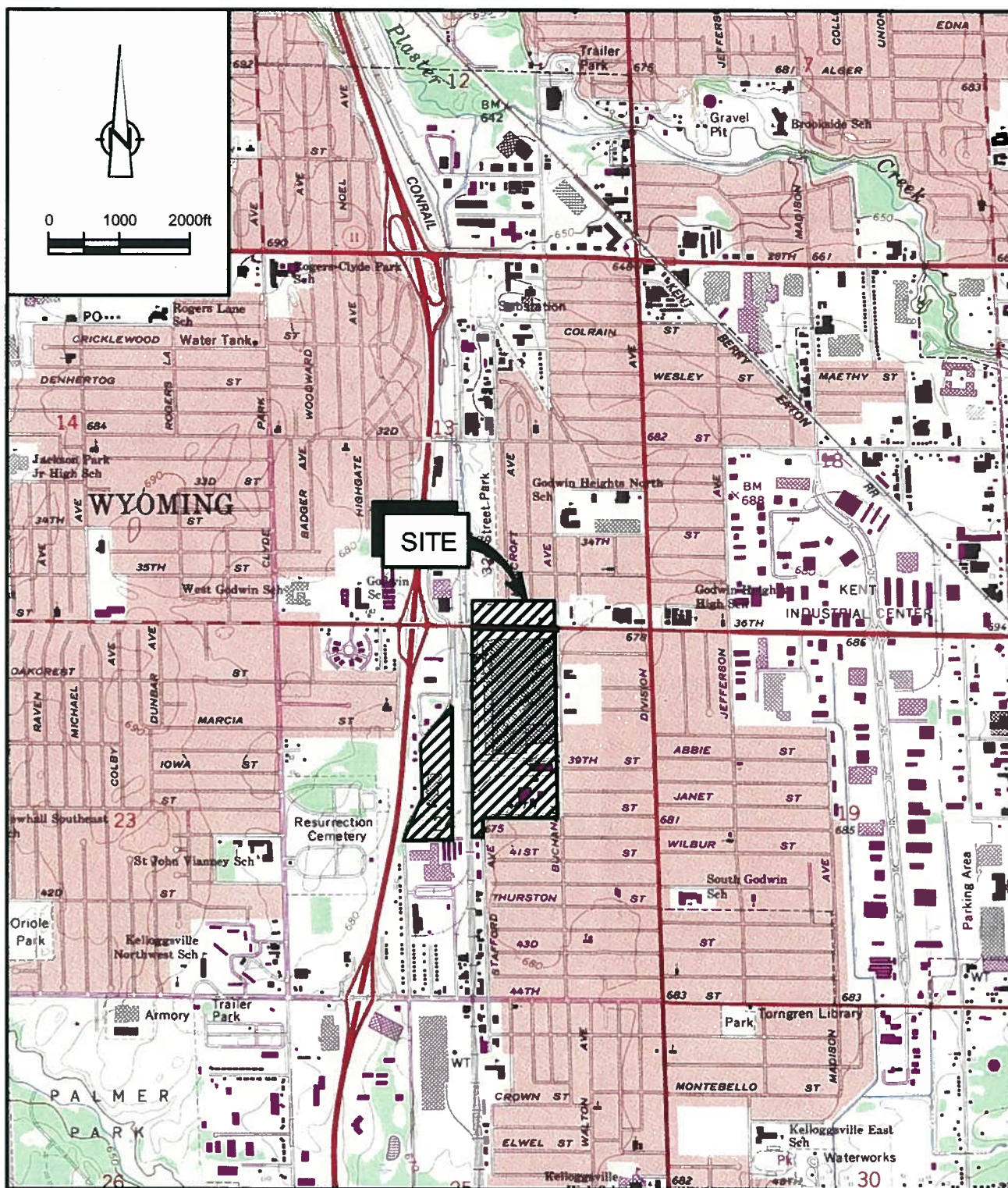
6.0 REPORTING

Upon completion of the field activities, receipt of final analytical results, and data quality assessment and validation, a report summarizing the results will be generated.

7.0 SCHEDULE

An access agreement for the off-Site locations will be negotiated with the City of Wyoming (property owner). It is anticipated that field activities will be initiated in Spring 2009, dependent upon weather conditions and procurement of access to off-Site locations. A generalized proposed schedule for the implementation of the field activities and reporting is presented below. A more detailed schedule will be provided upon verification of start date.

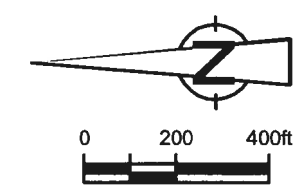
- Completion of Field Activities – Approximately 1 to 2 weeks from mobilization to completion.
- Submittal of Data Report – Approximately 8 to 12 weeks from completion of field activities.



SOURCE: USGS QUADRANGLE MAP;
GRAND RAPIDS WEST, MICHIGAN



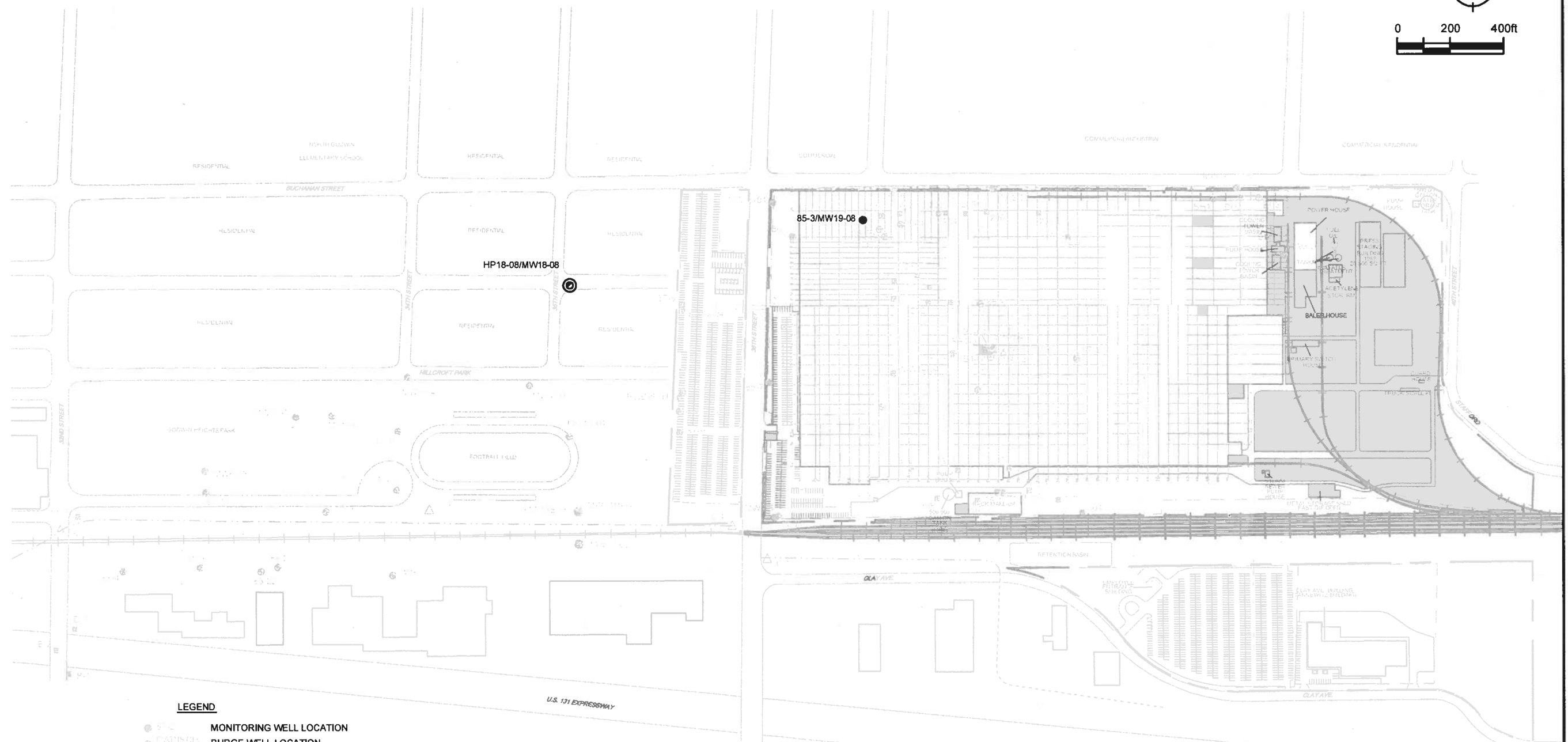
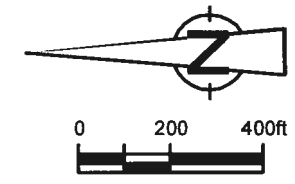
figure 1.1
SITE LOCATION
GRAND RAPIDS METAL PLANT
Wyoming, Michigan



- LEGEND**
- 87-2 MONITORING WELL LOCATION
 - ⊕ PW1-04 PURGE WELL LOCATION
 - 307 SOIL BORING LOCATION
 - ⊙ X-10 DESTROYED/REMOVED MONITORING WELL LOCATION
 - △ C-2 CULVERT LOCATION
 - ▲ 87-3 AIR PURGE WELL
 - ⊕ BH1 APPROXIMATE LOCATION OF DEWATERING WELLS
 - APPROXIMATE PROPERTY BOUNDARY
 - - - FENCE
 - + -+ -+ RAILROAD
 - - - COAL DRAIN



figure 1.2
SITE PLAN
GRAND RAPIDS METAL PLANT
Wyoming, Michigan



LEGEND

- 85-3 MONITORING WELL LOCATION
- ⊕ 85-3 PURGE WELL LOCATION
- ⊕ 85-3 SOIL BORING LOCATION
- ⊕ 85-3 DESTROYED/REMOVED MONITORING WELL LOCATION
- △ 85-3 CULVERT LOCATION
- △ 85-3 AIR PURGE WELL
- ⊕ 85-3 APPROXIMATE LOCATION OF DEWATERING WELLS
- ⊕ 85-3 PROPOSED HYDROPUNCH/MONITORING WELL LOCATION
- APPROXIMATE PROPERTY BOUNDARY
- FENCE
- RAILROAD
- COLE DRAIN

figure 3.1

**PROPOSED HYDROPUNCH AND MONITORING WELL LOCATIONS
GRAND RAPIDS METAL PLANT
Wyoming, Michigan**



TABLE 3.1
SAMPLING AND ANALYSIS PLAN
2008 GROUNDWATER INVESTIGATION WORK PLAN
GENERAL MOTORS CORPORATION
GRAND RAPIDS METAL PLANT
WYOMING, MICHIGAN

<i>Proposed Investigation Activity</i>	<i>Reason for Activity</i>	<i>Quantity of Sample Locations</i>	<i>Quantity of Samples</i>	<i>Sample Matrix</i>	<i>Sample Interval</i>	<i>Analysis</i>
Vertical Aquifer Sampling	Vertical/horizontal delineation	1	8 ⁽¹⁾	groundwater	discrete vertical intervals of the water bearing unit	TCL VOCs
Groundwater Sampling	Additional Groundwater Monitoring	3	3	groundwater	screened interval	TCL SVOCs
LNAPL Sampling	LNAPL Fingerprinting	1	1	LNAPL	--	TCL SVOCs, TCL VOCs. TAL metals, PCBs, density mean molecular weight
Collection of waste characterization samples	Disposal	1 ⁽²⁾	1 ⁽²⁾	soil	--	TCLP metals, VOCs, SVOCs, RCI, total VOCs
		1 ⁽²⁾	1 ⁽²⁾	wastewater	--	TCLP metals, VOCs, SVOCs, RCI, total VOCs

Notes:

⁽¹⁾ Number of samples includes Quality Assurance/Quality Control samples.

⁽²⁾ Number of samples to be determined in the field.

TCL - Target Compound List

VOCs - Volatile Organic Compounds

TCLP - Toxicity Characteristic Leaching Procedure

SVOCs - Semi-volatile Organic Compounds

TAL - Target Analyte List

RCI - Reactivity, Corrosivity, and Ignitability

APPENDIX A

QUALITY ASSURANCE PROJECT PLAN

APPENDIX A

QUALITY ASSURANCE PROJECT PLAN (QAPP)

**General Motors Corporation
Grand Rapids Metal Plant
Wyoming, Michigan**

**AUGUST 2008
REF. NO. 017360 (16)**

QUALITY ASSURANCE PROJECT PLAN (QAPP)

GENERAL MOTORS CORPORATION
GRAND RAPIDS METAL PLANT
WYOMING, MICHIGAN
REVISION NUMBER 0
JULY 15, 2008

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TABLE OF CONTENTS

	<u>Page</u>
1.0 PROJECT DESCRIPTION.....	1
1.1 INTRODUCTION	1
1.1.1 PROJECT OBJECTIVES AND DECISION STATEMENT	1
1.1.2 PROJECT STATUS/PHASE	2
1.1.3 QAPP PREPARATION GUIDELINES	2
1.2 SITE DESCRIPTION	2
1.3 SITE HISTORY	3
1.4 PROJECT OBJECTIVES AND INTENDED DATA USE	3
1.4.1 TARGET PARAMETER LIST	3
1.4.1.1 FIELD PARAMETERS	3
1.4.1.2 LABORATORY PARAMETERS	4
1.5 SAMPLING LOCATIONS	4
2.0 PROJECT ORGANIZATION AND RESPONSIBILITIES	1
2.1 MANAGEMENT RESPONSIBILITIES	1
2.2 QUALITY ASSURANCE RESPONSIBILITIES	2
2.3 LABORATORY RESPONSIBILITIES	3
2.4 FIELD RESPONSIBILITIES	4
3.0 QUALITY ASSURANCE OBJECTIVES FOR MEASUREMENT DATA	1
3.1 PRECISION	1
3.1.1 DEFINITION	1
3.1.2 FIELD PRECISION SAMPLE OBJECTIVES	1
3.1.3 LABORATORY PRECISION SAMPLE OBJECTIVES	2
3.2 ACCURACY	2
3.2.1 DEFINITION	2
3.2.2 FIELD ACCURACY OBJECTIVES	2
3.2.3 LABORATORY ACCURACY SAMPLE OBJECTIVES	3
3.3 REPRESENTATIVENESS	3
3.3.1 DEFINITION	3
3.3.2 MEASURES TO ENSURE REPRESENTATIVENESS OF FIELD DATA	4
3.3.3 MEASURES TO ENSURE REPRESENTATIVENESS OF LABORATORY DATA	4
3.4 COMPLETENESS	4
3.4.1 DEFINITION	4
3.4.2 FIELD COMPLETENESS OBJECTIVES	5
3.4.3 LABORATORY COMPLETENESS OBJECTIVES	5
3.5 COMPARABILITY	5
3.5.1 DEFINITION	5
3.5.2 MEASURES TO ENSURE COMPARABILITY OF FIELD DATA	5

TABLE OF CONTENTS (CONT'D)

	<u>Page</u>
3.5.3 MEASURES TO ENSURE COMPARABILITY OF LABORATORY DATA	6
3.6 LEVEL OF QUALITY CONTROL EFFORT	6
4.0 SAMPLING PROCEDURES	1
4.1 SAMPLE CONTAINERS	1
4.2 SAMPLE LABELING	1
4.3 FIELD QC SAMPLE COLLECTION	2
4.3.1 EQUIPMENT RINSATE BLANK SAMPLE COLLECTION	2
4.3.2 FIELD DUPLICATE SAMPLE COLLECTION	2
4.3.2.1 WATER SAMPLES	2
4.3.2.2 SOIL SAMPLES	2
4.3.3 MS/MSD SAMPLE COLLECTION	2
5.0 CUSTODY PROCEDURES	1
5.1 FIELD CUSTODY PROCEDURES	1
5.1.1 FIELD PROCEDURES	2
5.1.2 TRANSFER OF CUSTODY AND SHIPMENT PROCEDURES	3
5.2 LABORATORY CHAIN-OF-CUSTODY PROCEDURES	4
5.3 STORAGE OF SAMPLES	5
5.4 FINAL PROJECT FILES CUSTODY PROCEDURES	5
6.0 CALIBRATION PROCEDURES AND FREQUENCY	1
6.1 FIELD INSTRUMENT CALIBRATION PROCEDURES	1
6.2 LABORATORY INSTRUMENT CALIBRATION PROCEDURES	1
7.0 ANALYTICAL PROCEDURES	1
7.1 FIELD ANALYTICAL PROCEDURES	1
7.2 LABORATORY ANALYTICAL PROCEDURES	1
7.2.1 LIST OF PROJECT TARGET COMPOUNDS AND LABORATORY DETECTION LIMITS	1
7.2.2 LIST OF METHOD SPECIFIC QUALITY CONTROL (QC) CRITERIA	2
8.0 INTERNAL QUALITY CONTROL CHECKS	1
8.1 FIELD QUALITY CONTROL	1
8.1.1 EQUIPMENT RINSATE BLANKS	1
8.1.2 TRIP BLANKS	1
8.1.3 FIELD DUPLICATE SAMPLES	2
8.2 LABORATORY PROCEDURES	2

TABLE OF CONTENTS (CONT'D)

	<u>Page</u>
9.0 DATA REDUCTION, VALIDATION AND REPORTING	1
9.1 DATA REDUCTION	1
9.1.1 FIELD DATA REDUCTION PROCEDURES	1
9.1.2 LABORATORY DATA REDUCTION PROCEDURES	1
9.2 DATA VALIDATION	2
9.2.1 PROCEDURES USED TO EVALUATE FIELD DATA	2
9.2.2 PROCEDURES TO VALIDATE LABORATORY DATA	2
9.3 DATA REPORTING	4
9.3.1 FIELD DATA REPORTING	4
9.3.2 LABORATORY DATA REPORTING	4
10.0 PERFORMANCE AND SYSTEM AUDITS	1
10.1 FIELD PERFORMANCE AND SYSTEM AUDITS	1
10.1.1 INTERNAL FIELD AUDIT RESPONSIBILITIES	1
10.1.2 EXTERNAL FIELD AUDIT RESPONSIBILITIES	1
10.2 LABORATORY PERFORMANCE AND SYSTEM AUDITS	2
10.2.1 INTERNAL LABORATORY AUDIT RESPONSIBILITIES	2
10.2.2 EXTERNAL LABORATORY AUDIT RESPONSIBILITIES	2
11.0 PREVENTATIVE MAINTENANCE	1
11.1 FIELD INSTRUMENT PREVENTATIVE MAINTENANCE	1
11.2 LABORATORY INSTRUMENT PREVENTATIVE MAINTENANCE	1
12.0 SPECIFIC ROUTINE PROCEDURES USED TO ASSESS DATA PRECISION, ACCURACY, AND COMPLETENESS	1
12.1 FIELD MEASUREMENTS	1
12.2 LABORATORY DATA	1
12.2.1 PRECISION	2
12.2.2 ACCURACY	2
12.2.3 COMPLETENESS	2
12.3 STATISTICAL EVALUATIONS	2
12.3.1 PERCENT RECOVERY	2
12.3.2 RELATIVE PERCENT DIFFERENCE	3
13.0 CORRECTIVE ACTION	1
13.1 FIELD CORRECTIVE ACTION	1
13.2 LABORATORY CORRECTIVE ACTION	1
13.3 CORRECTIVE ACTION DURING DATA VALIDATION AND DATA ASSESSMENT	2

TABLE OF CONTENTS (CONT'D)

	<u>Page</u>
14.0 QUALITY ASSURANCE (QA) REPORTS	1
15.0 REFERENCES	1

LIST OF TABLES

TABLE 1.1	SUMMARY OF SAMPLING AND ANALYSIS PROGRAM
TABLE 1.2	VERTICAL AQUIFER AND MONITORING WELL PARAMETER LIST
TABLE 1.3	LNAPL PARAMETER LIST
TABLE 1.4	SOIL PARAMETER LIST
TABLE 1.5	WASTE CHARACTERIZATION PARAMETER LIST
TABLE 4.1	SAMPLE CONTAINER, PRESERVATION, SHIPPING, AND PACKAGING REQUIREMENTS
TABLE 7.1	SUMMARY OF ANALYTICAL METHODS

ATTACHMENTS

ATTACHMENT 1	FIELD ANALYTICAL STANDARD OPERATING PROCEDURES
ATTACHMENT 2	LABORATORY ANALYTICAL STANDARD OPERATING PROCEDURES
ATTACHMENT 3	LABORATORY REFERENCE DATA
ATTACHMENT 4	FIELD AUDIT CHECKLIST
ATTACHMENT 5	LABORATORY AUDIT CHECKLIST

1.0 PROJECT DESCRIPTION

The following outlines the scope of the investigation and monitoring activities to be performed at the General Motors Corporation (GM) Grand Rapids Metal Plant located 300 36th Street in Wyoming, Michigan (Site).

This Quality Assurance Project Plan (QAPP) presents the organization, objectives, planned activities, and specific quality assurance / quality control (QA/QC) procedures associated with the proposed investigation 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.

This QAPP addresses the QA/QC elements in the U.S. EPA Region 5 QAPP policy presented in the "U. S. EPA RCRA QAPP Instructions" dated April 1998 and other relevant guidance documents. The QA/QC procedures described in this QAPP are consistent with U.S. EPA guidance in general.

1.1 INTRODUCTION

This QAPP has been prepared on behalf of GM. The QAPP is a component of the 2008 Groundwater Investigation Work Plan (Work Plan) for the Site. In addition, the QAPP is a component of the 2008 Bulk Unload Area Work Plan and the 2005 Groundwater Monitoring Work Plan.

1.1.1 PROJECT OBJECTIVES AND DECISION STATEMENT

The primary objectives for data collection activities include:

- i) Further evaluate and delineate chlorinated volatile organic compound (VOC) impacts to groundwater at the Site above the Michigan Act 451, Part 201 Residential Cleanup Criteria;
- ii) Further define hydrogeologic conditions at the Site;

- iii) Evaluate the nature of the light non-aqueous phase liquid (LNAPL) identified at the Site; and
- iv) Further evaluate and delineate polynuclear aromatic hydrocarbon (PNA), polychlorinated biphenyl (PCB), and metals impacts to soil at the Bulk Unload Area of the Site above the Michigan Act 451, Part 201 Residential Cleanup Criteria.

The Decision Statement for the investigation is as follows:

- i) Determine potential remedial actions that are required or sufficient to achieve Michigan Act 451, Part 201 Residential Cleanup Criteria.

Associated specific objectives for field and laboratory data collection are discussed in Section 1.4 of this plan.

1.1.2 PROJECT STATUS/PHASE

The project status and investigation approach is presented in the Work Plan. Section 1.1 summarizes the Site background. Section 3.0 describes the proposed scope of work for the investigation at the Site. Section 5.0 describes how the data evaluation procedures for this project will be conducted.

1.1.3 QAPP PREPARATION GUIDELINES

As previously noted, this QAPP has been prepared to present the project specific QA/QC elements in the "U.S. EPA Region 5 QAPP Instructions" dated April 1998.

1.2 SITE DESCRIPTION

The general Site description is provided and is incorporated here by reference to the following Work Plan Sections.

Location	Work Plan Section 1.0
Site Dimensions/Borders	Work Plan Section 1.1

Natural and Manmade Features	Work Plan Section 1.1
Topography	Work Plan Section 1.1
Local Geology and Hydrology	Work Plan Section 2.2

1.3 **SITE HISTORY**

The Site history is provided in Sections 1.1 and 2.1 of the Work Plan and incorporated here by reference.

General History	Work Plan Section 1.1
Past Data Collection Activities	Work Plan Section 1.1 and 2.1
Current Status	Work Plan Section 2.1

1.4 **PROJECT OBJECTIVES AND INTENDED DATA USE**

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 historical operations at the Site. The field and laboratory parameters are summarized below and presented in Table 1.1.

1.4.1.1 **FIELD PARAMETERS**

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 (groundwater), at a minimum, the following parameters will be determined with field testing equipment: pH, specific conductivity, and temperature. The low-flow purging techniques utilized for groundwater sampling, will include additional field parameters including temperature, pH, conductivity, turbidity, dissolved oxygen, and oxidation/reduction potential (ORP).

1.4.1.2 LABORATORY PARAMETERS

The laboratory parameters for groundwater samples include target compound list (TCL) VOCs and TCL semi-volatile organic compounds (SVOCs). The basis for the selected parameter list is provided in Section 5.0 of the Work Plan.

Table 1.2 presents the estimated quantitation limits (EQL) and method detection limits (MDL) for each compound or analyte for vertical aquifer and groundwater samples. MDL studies have been performed by Test America Laboratories (TestAmerica) in accordance with the procedures established in the Federal Register, Volume 49, number 209, October 26, 1984 pp. 198-199, the results of these MDL studies are presented in Attachment 3.

In addition to the groundwater sampling, LNAPL samples will be analyzed for TCL VOCs, TCL SVOCs, Site-specific Target Analyte List (TAL) metals less earth metals (aluminum, calcium, iron, magnesium, potassium, and sodium), polychlorinated biphenyls (PCBs), hydrocarbon fuel scan, average molecular weight, dynamic viscosity, and density, presented with EQLs in Table 1.3; soil samples from the Bulk Unload Area will be analyzed for Site-specific TAL metals PCBs, and PNAs, presented with EQLs in Table 1.4; and waste characterization will be completed on soil cuttings and purge/decontamination water as presented with EQLs in Table 1.5.

1.5 SAMPLING LOCATIONS

The Work Plan provides a summary and rationale for the location of groundwater samples at the Site. It is possible, however, that depending on the nature of encountered field conditions, sampling locations may change. The person responsible for making such decisions will be the Field Quality Assurance (QA) Officer whose responsibilities are described in Section 2.0 of this QAPP. Any change of the sampling strategy will only be implemented after approval from the GM Project Manager.

2.0 PROJECT ORGANIZATION AND RESPONSIBILITIES

The GM Project Manager will have the primary responsibility for the implementation of the Work Plan. TestAmerica's laboratory in North Canton, Ohio (TestAmerica) will perform the analyses of environmental samples collected at the Site with support from their Portland, Oregon Laboratory and Friedman & Bruya, Inc. of Seattle, Washington. This section defines the roles and responsibilities of the individuals who will perform the Work Plan activities.

2.1 MANAGEMENT RESPONSIBILITIES

A description of the key project personnel responsibilities is presented in the following sections. Management responsibilities are as follows:

Kurt Blizzard – GM Project Manager

The GM Project Manager is responsible for implementing the project, and has the authority to commit the resources necessary to meet project objectives and requirements. The GM Project Manager's primary function is to ensure that technical, financial, and scheduling objectives are achieved successfully. The GM Project Manager will provide the major point of contact and control matters concerning the project and represent the project team at regulatory agency meetings and public hearings. The Project Manager will define project objectives and develop a detailed project schedule. The GM 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.

Jennifer Quigley – CRA Project Manager

The Project Manager is responsible for managing the implementation of the 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.

Kevin Kowalk - CRA Project Coordinator

The Project Coordinator will assist the Project Manager in day-to-day project management. The Project Coordinator is responsible for coordinating all field activities and the procurement of project subcontractors. Additional responsibilities include:

- i) Assisting in monitoring the progress and quality;
- ii) Preparing and reviewing reports; and
- iii) Providing technical support of project activities.

2.2 QUALITY ASSURANCE RESPONSIBILITIES

The Quality Assurance team will consist of a Quality Assurance Officer and the Project Manager. Quality Assurance responsibilities are described as follows:

Paul Wiseman – CRA Quality Assurance (QA) Officer

The QA Officer reports directly to the Encore Project Manager and will be responsible for ensuring that all QA/QC procedures are being followed. The QA Officer will be responsible for overseeing the review of all field and laboratory data. Additional responsibilities include:

- i) Assuring the application and effectiveness of the QAPP by the analytical laboratory and the project staff;
- ii) Conducting internal QA/QC of the investigation activities;
- iii) Providing input to the Project Manager and the Project Engineer as to corrective actions required resulting from the above-mentioned evaluations;
- iv) Preparation and review of data validation and audit reports; and
- v) 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. The QA Officer will monitor the performance of the laboratory to ensure that the Data Quality Objectives for the project are met.

2.3 LABORATORY RESPONSIBILITIES

TA will provide laboratory services in support of the Work Plan.

Denise Heckler – TestAmerica Laboratory Project Manager

The Laboratory Project Manager will report directly to the CRA QA Officer and will be responsible for ensuring all resources of the laboratory are available on an as-required basis through coordination with the Operations Manager. The Laboratory Project Manager will also be responsible for the approval of the final analytical reports and approval of the laboratory's ability to adhere to the QAPP. The Laboratory Project Manager will sign all final laboratory analytical reports provided from the analysis of the Investigation samples.

Raymond Risdon – TestAmerica Laboratory Operations Manager

The Laboratory Operations Manager will report to the Project Manager and 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

Dorothy Leeson – TestAmerica Laboratory QA Officer

The Laboratory QA Officer is responsible for coordination and oversight of the laboratories quality assurance program which includes:

- i) Coordinate and oversee laboratory system audits;
- ii) Overview of QA/QC documentation;
- iii) Conduct detailed data review upon request;
- iv) Implement and document laboratory corrective actions, if required;
- v) Technical representation of laboratory QA procedures;
- vi) Oversee preparation of laboratory SOPs; and
- vii) Approval of the QAPP.

John McFadden – TestAmerica Laboratory Sample Receiving Group Leader

The Laboratory Sample Receiving Group Leader will report to the Laboratory Operations Manager and will be responsible for overseeing the sample custodians in the following:

- i) Receiving and inspecting the incoming sample containers;
- ii) Recording the condition of the incoming sample containers;
- iii) Signing appropriate documents;
- iv) Verifying chain-of-custody and its correctness;
- v) Notifying the Project Manager and Operations Manager of sample receipt and inspection;
- vi) Assigning a unique identification number and customer number, and entering each into the sample receiving log;
- vii) Initiating transfer of samples to lab sections; and
- viii) Controlling and monitoring 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 (SOPs).

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 9.2.2 of this document and be presented in a formal written report for submittal to the CRA Project Manager.

2.4 FIELD RESPONSIBILITIES

Kevin Kowalk – CRA 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 Project Manager. The Field QA Officer works with the project Health & Safety Officer to conduct operations in compliance with the project Health & Safety Plan. The Field QA Officer will facilitate communication and coordinate efforts between the Project Manager and the field team members. Other responsibilities include:

- i) Developing and implementing field-related work plans, ensuring schedule compliance, and adhering to management-developed project requirements;
- ii) Coordinating and managing field staff, including sampling and drilling;
- iii) Performing field system audits;
- iv) Overseeing quality control for technical data provided by the field staff;
- v) Preparing, and approving of text and graphics required for field team efforts;
- vi) Coordinating and overseeing technical efforts of subcontractors assisting the field team;
- vii) Identifying problems in the field, resolving difficulties in consultation with the, Project QA Officer and CRA Project Manager, implementing and documenting corrective action procedures;
- viii) Approving the QAPP; and
- ix) Participating in preparation of the final reports.

Field Team Personnel

Field Team Personnel involved in the investigation will be responsible for:

- i) Performance of field activities as detailed in the Work Plan and in compliance with the DQO outlined in this QAPP; and
- ii) Immediately reporting any accidents and/or unsafe conditions to the Site Health & Safety Officer and taking all reasonable precautions to prevent injury.

3.0 QUALITY ASSURANCE OBJECTIVES FOR MEASUREMENT DATA

The 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 field and analytical methods. The overall QA objective for measurement data is to develop and implement procedures for field sampling, chain-of-custody, lab analyses, and reporting that will provide results which are adequate for supporting the Work Plan objectives and legally defensible in a court of law. The QAPP program will include:

- i) A mechanism for ongoing control of measurements and evaluation of data quality.
- ii) A measure of data quality in terms of precision, accuracy, representativeness, completeness, and comparability.

The following section is a general discussion of the criteria used to measure the field and laboratory analytical data quality. The laboratory reference data and SOPs provided in Attachments 2 and 3, present the performance criteria for laboratory QC objectives.

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 stated in terms of relative percent difference (RPD). The overall precision of measurement data is a mixture of sampling and analytical factors. 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. The analytical results from the field duplicate samples will provide data on sampling precision. The results from the laboratory created duplicate samples 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). The analytical data will be presented in summary table format. The precision criteria for laboratory MS/MSD analyses are provided in the laboratory reference data provided in Attachment 3.

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 field equipment rinsate and trip blanks. Field equipment rinsate and trip blanks will be collected as appropriate for each sampling effort.

Field equipment rinsate blanks will be collected by passing laboratory purified water over and/or through the respective field equipment utilized during each sampling effort. One rinsate blank will be collected every 20 investigative samples. However, at a minimum one equipment rinsate blank sample will be collected per day for each type of field equipment used for the sampling effort. Field rinsate 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 rinsate samples may not be collected as part of that field effort.)

Trip blank samples will be supplied by the laboratory and submitted with each cooler that includes VOC analysis containers. Trip blank samples will be analyzed for each VOC for which environmental media have been preserved in the field 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 (1) set of MS/MSD analyses will be performed with each batch of twenty (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 MDLs and EQLs 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 laboratory analytical reports. Current acceptance criteria are presented in the laboratory reference data provided in Attachment 3.

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 and are evaluated against the acceptance criteria presented in laboratory analytical reports. Current acceptance criteria are presented in the laboratory reference data provided in Attachment 3.

3.3 REPRESENTATIVENESS

3.3.1 DEFINITION

Representativeness expresses the degree to which sample data accurately and precisely represents 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 be biased; that is, sampling associated with the soil and groundwater will be based on the observed presence/absence of site specific contaminants, and/or site knowledge. Specific sampling technique descriptions, which allow consistency, repetitiveness and thus representativeness, are provided in the Work Plan.

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. Field completeness objective for this project will be 90 percent.

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 completeness objective will be 90 percent.

3.5 COMPARABILITY

3.5.1 DEFINITION

Comparability is a qualitative parameter expressing the confidence with which one data set can be compared with another.

3.5.2 MEASURES TO ENSURE COMPARABILITY OF FIELD DATA

Sample data should be comparable with other measurement data for similar samples and sample conditions. This goal is achieved through using standard operating procedures to collect, preserve, store, and analyze representative samples and the reporting of analytical results. The field procedures for the various activities to be conducted during this investigation provide guidelines to generate reproducible results.

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 or mass of environmental media.

- i) Solid Matrices - micrograms (μg) contaminant per kilogram (kg) for organic analyses, and milligrams (mg) contaminant per kilogram (kg) for inorganic analyses of media (Dry Weight).
- ii) Aqueous Matrices – micrograms (μg) contaminant per liter (L) of media for organic analyses, and milligrams (mg) per liter (L) for inorganic analyses.
- iii) Non-Aqueous Phase Liquids – (NAPL) – micrograms (μg) contaminant per kilogram (kg) of media.

Additional guidance on analytical data reporting is contained in the laboratory SOPs.

3.6 LEVEL OF QUALITY CONTROL EFFORT

Equipment rinsate, trip, and method blanks samples, field duplicate samples, laboratory control and MS/MSD samples will be prepared and analyzed to determine the data quality provided by the sampling and analysis activities conducted during the execution of the investigation program.

Equipment rinsate 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 rinsate blank samples will be analyzed to check for contamination of equipment introduced during sampling at the Site. One equipment rinsate blank will be collected for every 20 investigative samples. However, a minimum of one will be collected each day of sampling activities for each type of non-dedicated or non-disposable equipment.

Trip blanks are used to assess the potential for contamination during sample storage and shipment. The trip blank consists of purified water that has been purchased by the

laboratory and provided with the sample containers to be used for the sampling of 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 during the analytical process.

Field duplicate samples are analyzed to check for sampling and analytical reproducibility. One field duplicate will be collected for every 20 or fewer investigative samples collected per day of sampling activities.

Matrix spikes will provide information to assess the precision and accuracy of the analysis of the target parameters within the environmental media collected at the Site. Matrix spikes will be performed in duplicate for all TCL/TAL parameters. One MS/MSD will be collected for every 20 or fewer investigative samples per sample matrix (i.e. soil, groundwater). Soil MS/MSD samples require a triple sample volume for VOCs only. Aqueous MS/MSD samples require triple the normal sample volume for VOCs analysis and double the volume for the remaining parameters.

4.0 SAMPLING PROCEDURES

Samples of groundwater, soil, and LNAPL will be obtained during the investigation program. The sampling procedures for the investigation will be consistent for the objectives of the project. The Work Plan describes each of the sampling tasks and objectives.

Refer to the Work Plan for the field investigation standard operating procedures.

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 4.1.

4.2 SAMPLE LABELING

Each sample will be labeled with a unique sample number that will facilitate tracking and cross-referencing of sample information. Field blank and field duplicate samples also will be numbered with a unique sample number to prevent analytical bias of field QC samples.

Example: S-17360-MMDDYY-XX-001

S	- designates types of sample (S-soil, GW-groundwater)
17360	- Site reference number
MMDDYY	- designates date of collection presented as month/day/year
XX	- sampler's initials
01	- sequential number starting with 001 at the start of the project

Field blank and field duplicate samples also will be numbered with a unique sample number, consistent with the numbering system described above, to prevent laboratory bias of field QC samples.

4.3 FIELD QC SAMPLE COLLECTION

4.3.1 EQUIPMENT RINSATE BLANK SAMPLE COLLECTION

Equipment rinsate blank samples will be collected when non-dedicated or non-disposable sampling equipment is used to collect samples. Equipment rinsate blanks consist of laboratory purified 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, which are 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 concurrently with the investigative sample alternating the filling of each sample container using the procedures identified in the Work Plan.

4.3.2.2 SOIL SAMPLES

Soil field duplicates, if necessary, will be collected concurrently with the investigative sample using the procedures identified in the Work Plan.

4.3.3 MS/MSD SAMPLE COLLECTION

MS/MSD sample collection for aqueous samples requires triple the sample volume for all analysis. The sampling procedure specified in Section 4.3.2.1 is used to collect aqueous samples. Soil MS/MSD samples require triple volume for VOC analyses. No additional

volume is required for remaining soil parameters. Section 4.3.2.2 describes the sampling procedures for soil sample collection.

5.0 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 evidence files. Final evidence files, including all originals of laboratory reports, are maintained under document control in a secure area.

Custody of a sample begins when it is collected by or transferred to an individual and ends when that individual relinquishes or disposes of the sample. A sample or evidence file is under your custody if:

1. the item is in actual possession of a person;
2. the item is in the view of the person after being in actual possession of the person;
3. the item was in actual possession but is stored to prevent tampering; or
4. the item is in a designated and identified secure area.

5.1 FIELD CUSTODY PROCEDURES

Field personnel will be required to keep written records of field activities on applicable preprinted field forms or in a bound field notebook. The logbooks 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. Field forms and notebooks will be periodically reviewed by the Field QA Officer. Each member of the field team will be assigned a logbook. Each logbook title page should include field team member's name, project name, project start date, project end date, and unique logbook number.

The beginning of each entry in the logbook will contain the following information:

- i) Date;
- ii) Start time;

- iii) Weather;
- iv) Names of field personnel (including subcontractors);
- v) Level of personal protection used at the Site; and
- vi) Names of all visitors and the purpose of their visit.

For each measurement and sample collected the following will be recorded:

- i) Detailed description of sample point;
- ii) Equipment used to collect sample or make measurement and the date equipment was last calibrated;
- iii) Time sample was collected;
- iv) Sample description;
- v) Depth sample was collected;
- vi) Volume and number of containers; and
- vii) Sampler identification.

5.1.1 FIELD PROCEDURES

The 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:

- i) 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. The sample container, preservation methods, shipping, and packaging requirements are presented in Table 4.1.
- ii) Samples will be assigned a unique sample number and will be affixed to a sample label. The information to be placed on the sample label will include the sample ID number, the sample type, the sampler's name, date collected, preservation technique, and analytical parameter and method to be performed. Information on the labels will be completed with a ballpoint pen or indelible marker.

- iii) Samples are collected in pre-preserved sample containers supplied by the laboratory to minimize loss of the constituent(s) of interest due to physical, chemical or biological mechanisms.
- iv) Appropriate volumes will be collected to ensure that the EQLs can be successfully achieved and that the required QC Sample Analyses can be completed.

5.1.2 TRANSFER OF CUSTODY AND SHIPMENT PROCEDURES

- i) A chain-of-custody (COC) record will be completed during sample collection and will accompany each shipment identifying the contents of the shipment. The COC record will accompany the 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. As few people as possible should handle the samples.
- ii) 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.
- iii) Samples will also be transported in containers (coolers) packed with ice to maintain the temperatures prescribed in Table 4.1. Samples will be packaged for shipment and shipped to the appropriate laboratory for analysis with a separate signed chain-of-custody record enclosed in each sample cooler. VOC samples will be placed in foam sleeves that contain three containers per sleeve. 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. All samples will be placed in an upright position and limited to one layer of samples per each 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.
- iv) When samples are split with the U.S. EPA and Site representatives, a separate chain-of-custody will be prepared for the samples and marked to indicate to whom the samples are being split. The person relinquishing the samples to the U.S. EPA or the Site will require the representative's signature acknowledging sample receipt.

- v) 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 permanent documentation. Commercial carriers are not required to sign the custody record as long as the custody record is sealed inside the sample cooler and the custody tape remains intact.
- vi) 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 full-time sample receiving group leader supervises the sample receiving group who are assigned the responsibility of sample control. It will be the responsibility of the sample receiving group to receive all incoming samples. Once received, the custodian will document that the custody tape on the coolers is unbroken, that each sample is received in good condition (i.e., unbroken, cooled, etc.), that the associated paperwork, such as chain-of-custody forms have been completed and 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

Sample containers with volume remaining will be returned to secure and limited access storage. Upon completion of all laboratory analyses for each sample submittal and generation of the laboratory report, samples will be stored 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 all Federal, State, and local requirements.

Laboratory custody procedures and document control for those samples analyzed by the project laboratory will be carried out using the laboratory's standard operating procedures.

5.4 FINAL PROJECT FILES CUSTODY PROCEDURES

The final project file will be the central repository for all documents with information relevant to sampling and analysis activities as described in this QAPP. The 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 secured, limited access area and under custody of the CRA Project Manager or his designee.

The final project file will include:

- i) Project plans and drawings;
- ii) Field data records;
- iii) Sample identification documents and soil boring/monitoring well logs;
- iv) All chain-of-custody documentation;
- v) Correspondence;
- vi) References, literature;
- vii) Laboratory data deliverables;
- viii) Data validation and assessment reports;
- ix) Progress reports, QA reports; and
- x) 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 (5) years at which time the laboratory will contact CRA regarding the disposition of the project related files.

6.0 CALIBRATION PROCEDURES AND FREQUENCY

This section describes procedures for maintaining the accuracy for all the instruments and measurement equipment, which will be used for conducting field tests and laboratory analyses. These instruments and equipment will be calibrated prior to each use or according to a periodic schedule.

6.1 FIELD INSTRUMENT CALIBRATION PROCEDURES

Instruments and equipment used to gather, generate, or measure environmental data will be calibrated as specified in the field SOPs.

The field instruments are likely to include dissolved oxygen meters, pH meters, turbidity meters, specific conductance meters and PIDs. Field instruments will be used for real-time sample measurement during monitoring well sampling and organics screening for both on-site screening of soil samples and for health and safety air monitoring, as described in the Work Plan. On-site air monitoring for health and safety purposes and the screening of soil samples may be accomplished using PIDs.

Field instruments will be calibrated prior to use and the calibration will be verified on a daily basis as applicable. Specific information concerning calibration frequency, acceptance criteria, and the conditions that require more frequent calibration is contained in the field SOPs in Attachment 1.

Satisfactory completion of the pre-operation inspection will be noted on the Field Sampling Record, along with the results of each field measurement.

6.2 LABORATORY INSTRUMENT CALIBRATION PROCEDURES

Calibration procedures for a specific laboratory instrument will consist of initial calibration, initial calibration verification and continuing calibration verification. The SOPs in Attachment 2 present the specific calibration procedures for each method of analysis. The SOP for each analysis performed in the laboratory describes the calibration procedures, their frequency, acceptance criteria and the conditions that will require calibration. In all cases,

the initial calibration will be verified using an independently prepared calibration verification solution.

The use of materials of known purity and quality will be utilized for the analysis of environmental samples. The laboratory will carefully monitor the use of all laboratory materials including solutions, standards and reagents through well-documented procedures.

All solid chemicals and acids/bases used by the laboratory will be 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.

All materials including standards or standard solutions will be dated upon receipt, and will be identified by material name, lot number, purity or concentration, supplier, receipt/preparation date, recipient/preparer's name, expiration date and all other pertinent information.

7.0 ANALYTICAL PROCEDURES

Analytical procedures to be utilized for off-Site analysis of environmental samples will be based on referenced U.S. EPA analytical protocols and the laboratory SOPs that implement these methods. These project specific SOPs are provided in Attachment 2 to this QAPP.

7.1 FIELD ANALYTICAL PROCEDURES

Field analytical procedures include the measurement of pH/temperature, specific conductivity, dissolved oxygen, turbidity, and ORP during sampling of groundwater, and the qualitative measurement of VOC during the collection of soil samples at the Site. Specific guidance in the measurement of these parameters is presented in the SOPs provided in Attachment 1 of this QAPP.

7.2 LABORATORY ANALYTICAL PROCEDURES

Laboratory preparation and analytical SOPs and the methods upon which they are based are presented in Table 7.1. The referenced SOPs provided in Attachment 2 of this document are based on the U.S. EPA methodology requirements promulgated in:

- i) "Test Methods for Evaluating Solid Waste, Physical/Chemical Methods", SW-846 EPA, U.S. EPA Office of Solid Waste, 3rd Edition and promulgated updates, 1986; and
- ii) "Methods for Chemical Analysis of Water and Wastes," EPA-600/4-79-020, March 1983.

The EQLs for the target analytes and compounds for the environmental media to be analyzed as part of the investigation are presented in Table 1.2.

7.2.1 LIST OF PROJECT TARGET COMPOUNDS AND LABORATORY DETECTION LIMITS

A complete listing of project target compounds and project EQLs for each analyte group are listed in Tables 1.2, 1.3, 1.4, and 1.5. MDLs have been experimentally determined by the

project laboratory using the procedure identified in 40 CFR, Part 136 Appendix B. The laboratory's MDLs are provided in Attachment 3.

7.2.2 LIST OF METHOD SPECIFIC QUALITY CONTROL (QC) CRITERIA

The laboratory SOPs include a section that presents the minimum QC requirements for the project analyses. Section 8.0 references the frequency of the associated QC samples for each sampling effort and matrix.

8.0 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

QC procedures for pH/temperature, turbidity, conductivity, dissolved oxygen, and ORP, of water samples and PID screening soil samples will include calibrating the instruments as described in Section 6.1 of this QAPP, measuring duplicate samples and checking the reproducibility of the measurements by taking multiple readings on a single sample or the measurement reference standards. The QC information for field equipment is stated in Section 3.0 of this QAPP. Assessment of field sampling precision and bias will be made by collecting field duplicates and equipment rinsate blanks for laboratory analysis. Collection of the samples will be in accordance with the applicable procedures in Section 4.3 of this QAPP.

8.1.1 EQUIPMENT RINSATE BLANKS

Internal quality control checks will include analysis of equipment rinsate blanks to validate successful equipment cleaning activities. Whenever possible, dedicated equipment will be employed to reduce the possibility of cross-contamination of samples.

The frequency of equipment rinsate blank sample preparation will be one for every 20 investigative samples. However, at a minimum, one will be collected each day for each type of sampling equipment on which decontamination procedures have been performed.

8.1.2 TRIP BLANKS

Trip blanks samples will be supplied by the project laboratory using purified 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 which stores and transports project samples to be analyzed for VOCs along with the project samples.

8.1.3 FIELD DUPLICATE SAMPLES

Field duplicate samples are collected in a similar fashion to investigative samples at a minimum frequency of one duplicate per at least one in every 20 or fewer investigative samples by matrix at a minimum, one per sampling event. Field duplicate samples are analyzed by the laboratory to evaluate matrix, sample and analytical reproducibility.

8.2 LABORATORY PROCEDURES

The laboratories identified in Section 7.0 of this QAPP have QC programs to ensure the reliability and validity of the analysis performed at the laboratory. All analytical procedures are documented in writing as SOPs and each SOP includes a QC section, which addresses the minimum QC requirements for the procedure. The internal quality control checks may vary slightly for each individual procedure but in general will include the following QC requirements:

- i) Calibration Standards;
- ii) Instrument Performance Checks – Organics;
- iii) Initial and Continuing Calibration Checks;
- iv) Internal Standard Performance;
- v) Method Blank Samples;
- vi) Laboratory Control Samples and QC Check Samples;
- vii) Matrix Spike/Matrix Spike Duplicates;
- viii) Surrogates;
- ix) ICP Interference Check Samples (ICS);
- x) ICP Serial Dilution;
- xi) ICP and ICP/MS QC Analyses;
- xii) Reagent Checks; and
- xiii) Blind Check Samples.

All data will be properly recorded. The data package will include a summary of QC data as presented in Section 9.3.2. Any samples analyzed in nonconformance with QC criteria will be re-analyzed by the laboratory, if sufficient volume is available.

9.0 DATA REDUCTION, VALIDATION AND REPORTING

All data generated through in 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. Only direct read instrumentation will be employed in the field. The pH, conductivity, temperature, turbidity, dissolved oxygen, ORP, and PID readings collected in the field will be generated from direct read instruments following calibration per manufacturer's recommendations. Such data will be written into field logbooks immediately after measurements are taken. If errors are made, results will be legibly crossed out, initialed and dated by the field member, and corrected in a space adjacent to the original entry. Later, when the results forms required for this study are being filled out, the Project Coordinator will proof the forms to determine whether any transcription errors have been made by the field crew.

9.1.2 LABORATORY DATA REDUCTION PROCEDURES

For this project, the equations that will be employed in reducing data are found in the appropriate chapter of SW-846, Third Edition. Two of these equations, expressing analytical accuracy and precision, are presented in Section 3 of this QAPP. Such formulae make pertinent allowances for matrix type. All calculations are checked at the conclusion of each operating day. Errors are noted; corrections are made, but the original notations are 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 computer system. Data summaries will be reviewed by the department group leader or senior analyst. If approved, data are logged into the project database format. 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 Field QA Officer.

9.2.2 PROCEDURES TO VALIDATE LABORATORY DATA

The CRA QA Officer or designee will perform analytical data validation 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-99/008, October 1999 and the "U.S. EPA Contract Laboratory Program National Functional Guidelines for Inorganic Data Review ", EPA-540/R-94-013, February 1994 (CLP National Functional Guidelines).

The specific data qualifiers used will be as presented and defined in the CLP National Functional Guidelines. Data review and validation will consist of two tiers of assessment that incorporates an approach similar to "Innovative Approaches to Data Validation," U.S. EPA Region III, June 1995.

Tier I data validation will be performed on 100% of the laboratory data deliverables associated with the investigation and monitoring activities and will consist of the following components:

Organic Analysis

- i) technical holding times;
- ii) GC/MS instrument performance check;
- iii) initial and continuing calibration;
- iv) internal standard performance
- v) method, trip and field blanks;
- vi) system monitoring compounds (surrogate spikes);
- vii) MS/MSD results;
- viii) laboratory control samples; and
- ix) field duplicate samples.

Inorganic Analysis

- i) technical holding times;
- ii) initial calibration;
- iii) initial and continuing calibration verification;
- iv) blanks;
- v) ICP interference check samples;
- vi) ICP serial dilutions;
- vii) laboratory control samples;
- viii) MS/MSD and matrix duplicate results; and
- ix) field duplicate.

Tier II validation will be completed on 10 percent of the expanded deliverables during the investigation and monitoring activities. The Tier II validation will include the evaluation of all raw data and mass spectra to validate the above components as well as verify sample results.

The Data Validator will evaluate the completeness of each data package. 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 review will determine whether all required items are present and request copies of missing deliverables.

Samples submitted for waste characterization will be reported with reduced data packages and will not be validated.

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 associated with the investigation and monitoring activities will be sufficient to perform a data validation in accordance with protocols described in Section 9.2.2. The task of reporting laboratory data by GM will be initiated after the completion of data validation.

The laboratory Project Manager will perform a final review of the QC data summary packages and case narratives to determine whether the report meets the project requirements. In addition to the record of the chain-of-custody, the final laboratory data report format shall consist of the following expanded report deliverables:

- i) Title Page
 - project name and number;
 - laboratory project or lot number;
 - signature of the Laboratory QA Officer or his designee; and
 - date issued.
- ii) Table of Contents - laboratory report contents
- iii) Case Narrative
 - number of samples and respective matrices;;
 - laboratory analysis performed;
 - any deviations from intended analytical strategy;

- definition of data qualifiers used;
 - QC procedures utilized and references to the acceptance criteria;
 - 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; and
 - discussion of laboratory QC checks which failed to meet project criteria.
- iv) Analytical Methods Summary - methods of sample preparation and analyses for samples.
- v) Analytical Sample Summary - cross-reference table of laboratory sample to project sample identification numbers.
- vi) Shipping and Receiving Documents
- sample container documentation; and
 - sample reception information and original chain of custody record.
- vii) Chemistry Data Package by Analysis
- Sample Results
 - sample quantitation (report) limits (RL), reporting MDL and estimated values between the RL and MDL,
 - methods of sample preparation and analyses for samples,
 - raw data for sample results (dated chromatograms, parameter specific quantitation reports, mass spectra and instrument printouts;
 - QC Summary Data with Current Control Limits
 - matrix spike and matrix spike duplicate recoveries, laboratory control samples, method blank results, surrogate recoveries, GC/MS tuning results, and internal standards (organics),
 - matrix spike recoveries and matrix duplicate relative percent differences, laboratory control sample, serial dilutions, method blank results, and reagent blank results and interference check standards (inorganics);
 - Standard Data
 - initial calibration data, initial calibration checks, continuing calibration verification/check standards,

- initial and continuing calibration blanks,
- raw data for calibration data (dated chromatograms, parameter specific quantitation reports, mass spectra and instrument printouts;
- Raw QC Data - dated chromatograms, parameter specific quantitation reports, mass spectra and instrument printouts of QC samples;
- Miscellaneous Data
 - instrument run logs,
 - sample preparation records, and
 - instrument conditions.

Note: Only the TCL parameter list identified in Table 1.2 shall be included in the laboratory chromatographic quantitation reports for the investigation and monitoring well activities.

The final laboratory data reports associated with the waste characterization samples will include a reduced deliverable report. These laboratory data reports will not include standard data, QC and raw sample data and the miscellaneous data identified in the expanded deliverables report.

The project laboratory will provide electronic data deliverables (EDDs) in an EQuIS® 4-file format. The laboratory data will be downloaded into the EDDs directly from the laboratory information management system (LIMS). The EDDs are imported into EQuIS and the data are maintained in the database for manipulation and presentation.

The CRA QA Officer will verify the correctness of the analytical database by comparing the data from the database to the hardcopy analytical reports for a minimum of 10 percent of the sample results. If discrepancies between the database and hardcopy analytical reports are detected, a complete verification of the database will be performed.

10.0 PERFORMANCE AND SYSTEM AUDITS

A performance audit is an independently obtained 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 Field QA Officer. The audits will verify that all procedures are being followed. 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:

- i) Field sampling records, screening results, instrument operating records;
- ii) Sample collection;
- iii) Handling and packaging in compliance with procedures;
- iv) Maintenance of QA procedures; and
- v) Chain-of-custody reports.

Follow up audits will be conducted to correct deficiencies and to verify that procedures are maintained throughout the investigation. The field audit checklist is included in Appendix 4.

10.1.2 EXTERNAL FIELD AUDIT RESPONSIBILITIES

External audits may be conducted by the U.S. EPA Project Coordinator at any time during the field operations. These audits may or may not be announced and are at the discretion of the regulatory agency. The external field audits can include (but are not limited to) the following:

- i) Sampling equipment decontamination procedures;
- ii) Sample bottle preparation procedures;
- iii) Sampling procedures;

- iv) Examination of health and safety plans;
- v) Procedures for verification of field duplicates; and
- vi) 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 Project 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.

The performance audits will include submittal of blind performance evaluation samples along with project samples. The Project QA Officer will evaluate the analytical results to ensure the lab is maintaining acceptable QC performance. The laboratory audit checklist is provided in Attachment 5.

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 are 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 are 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 regulatory agency and may be conducted at least once prior to sampling and analysis activities.

External audits may include any of the following:

- i) Review of laboratory analytical procedures;
- ii) Laboratory on-site visits; and
- iii) 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:

- i) Sample receipt procedures;
- ii) Custody, sample security, and log-in procedures;
- iii) Review of instrument calibration logs;
- iv) Review of QA procedures;
- v) Review of log books;
- vi) Review of analytical SOPs; and
- vii) Personnel interviews.

A review of a data package from samples recently analyzed by the laboratory can include (but not be limited to) the following:

- i) Comparison of resulting data to the SOP or method;
- ii) Verification of initial and continuing calibrations within control limits;
- iii) Verification of surrogate recoveries and instrument timing results;
- iv) Review of extended quantitation reports for comparisons of library spectra to instrument spectra, where applicable; and
- v) Assurance that samples are run within holding times.

11.0 PREVENTATIVE MAINTENANCE

11.1 FIELD INSTRUMENT PREVENTATIVE MAINTENANCE

The field equipment preventative maintenance program ensures the effective completion of the sampling effort and is designed 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 PREVENTATIVE MAINTENANCE

Analytical instruments at the laboratory will undergo routine and/or preventative maintenance. The extent of the preventative maintenance will be a function of the complexity of the equipment.

Generally, annual preventative maintenance service will involve cleaning, adjusting, inspecting and testing procedures designed to deduce instrument failure and/or extend useful instrument life. Between visits, 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 can be made available upon request.

12.0 SPECIFIC ROUTINE PROCEDURES USED TO ASSESS DATA PRECISION, ACCURACY, AND COMPLETENESS

The following sections include the procedures and formulae utilized to assess the levels of precision, accuracy and completeness achieved during the associated sample analyses.

12.1 FIELD MEASUREMENTS

Field generated information such as pH, specific conductance data will be reviewed for validity. The review will be performed by the Field QA Officer and typically include 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 3.0 of this QAPP. The accuracy of pH specific conductance, dissolved oxygen, turbidity, and ORP, will be assessed using daily instrument calibration, calibration check, and blank data. Accuracy will be measured by determining the percent recovery of calibration check standards as defined in Section 12.2.2. Precision of the pH and specific conductance measurements will be assessed on the basis of the reproducibility of duplicate readings of a single sample and will be measured by determining the relative percent difference of the readings as defined in Section 12.2.1. 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:

$$\text{Completeness} = \frac{\text{Valid (usable) Data Obtained}}{\text{Total Data Planned}} \times 100$$

12.2 LABORATORY DATA

Laboratory results will be assessed for compliance with required precision, accuracy and completeness detailed in the following subsections:

12.2.1 PRECISION

The precision of laboratory analysis will be assessed by comparing the analytical results between MS/MSD analyses. The relative percent difference (RPD) will be calculated for each pair of duplicate analyses (laboratory MS/MSDs and field duplicates).

12.2.2 ACCURACY

The accuracy of laboratory results will be assessed for compliance with the established QC criteria that are described in Sections 3.0 and 8.0 of the QAPP using the analytical results of method blanks, reagent/preparation blank, SRMs or QC check samples and MS/MSD samples. The percent recovery (%R) of matrix spike samples and SRMs will be calculated.

12.2.3 COMPLETENESS

Completeness will be assessed by comparing the number of valid (usable) results (as determined by the QA Officer) to the total possible number of results using the formula presented in Section 12.1.

12.3 STATISTICAL EVALUATIONS

In the examination of data and determination of their precision and accuracy, standard statistical formulae will be used. Further details are provided in the following subsections

12.3.1 PERCENT RECOVERY

The percent recovery of a parameter is calculated by dividing the amount recovered by the true amount added and multiplying by 100. The percent recoveries of spiked samples are evaluated to establish the analytical accuracy of a measurement. Percent recovery is calculated using the following formula:

$$\%R = \frac{SSR - SR}{SA} \times 100$$

where:

SSR = Spiked Sample Result
SR = Sample Result or Background
SA = Spike Added

12.3.2 RELATIVE PERCENT DIFFERENCE

The relative percent difference (RPD) is calculated by dividing the absolute value of the difference between two numbers by their arithmetic mean and multiplying by 100. The RPD is used to evaluate the analytical precision of two replicate measurements (e.g., matrix spike/matrix spike duplicate). RPD is calculated using the following formula:

$$RPD = \frac{(R_1 - R_2)}{\frac{R_1 + R_2}{2}} \times 100$$

where:

R₁ = value of first result
R₂ = value of second result

13.0 CORRECTIVE ACTION

13.1 FIELD CORRECTIVE ACTION

Corrective action is intended to address problems that arise by identification, recommendation, approval, and implementation of measures that counter unacceptable procedures or deficient quality control performance. The Field QA Officer and CRA 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.

The corrective action procedures will be as follows:

- i) Identify/define the problem;
- ii) Assign responsibility for investigating the problem;
- iii) Investigate/determine the cause of the problem;
- iv) Determine an appropriate corrective action to eliminate the problem;
- v) Implement the corrective action;
- vi) Evaluate the effectiveness of the corrective action;
- vii) Verify that the corrective action has eliminated the problem;
- viii) Prepare a written record detailing the problem, corrective action utilized, and solution of the problem; and
- ix) Submit the Corrective Action Record (CAR) to the Task Coordinator who initiated the corrective action and the Project QA Officer, and Project Manager.

The above procedures may be implemented through the use of the Systems Audit as described previously. Any Field Team member of the project may initiate corrective action procedures by reporting in writing the nature of the suspected problem to the CRA Project Manager or CRA QA Officer. The CRA 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, CRA QA Officer, CRA Project Manager and GM Project Manager.

13.3 CORRECTIVE ACTION DURING DATA VALIDATION AND DATA ASSESSMENT

The CRA QA Officer 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/reanalysis of samples by the laboratory (if possible).

These actions are dependent upon the ability to mobilize the field team, whether the data to be collected is necessary to meet the required quality assurance objectives (e.g., the holding time for samples is not exceeded). When the CRA QA Officer identifies a corrective action situation, GM Project Manager will be responsible for approving the implementation of corrective action, including re-sampling, during data assessment. All corrective actions will be documented by the CRA Project Manager.

14.0 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, internal and external audits with responses;
- Performance evaluation sample results; internal and external sources; and
- Daily QA/QC exception reports/corrective actions.

QA/QC corrective action reports will be prepared by the CRA QA Officer when appropriate and presented to the project and/or laboratory management personnel so that performance criteria can be monitored for all analyses from each analytical department. The updated trend/QA charts prepared by the laboratory QA personnel will be distributed and reviewed by various levels of the laboratory management.

15.0 REFERENCES

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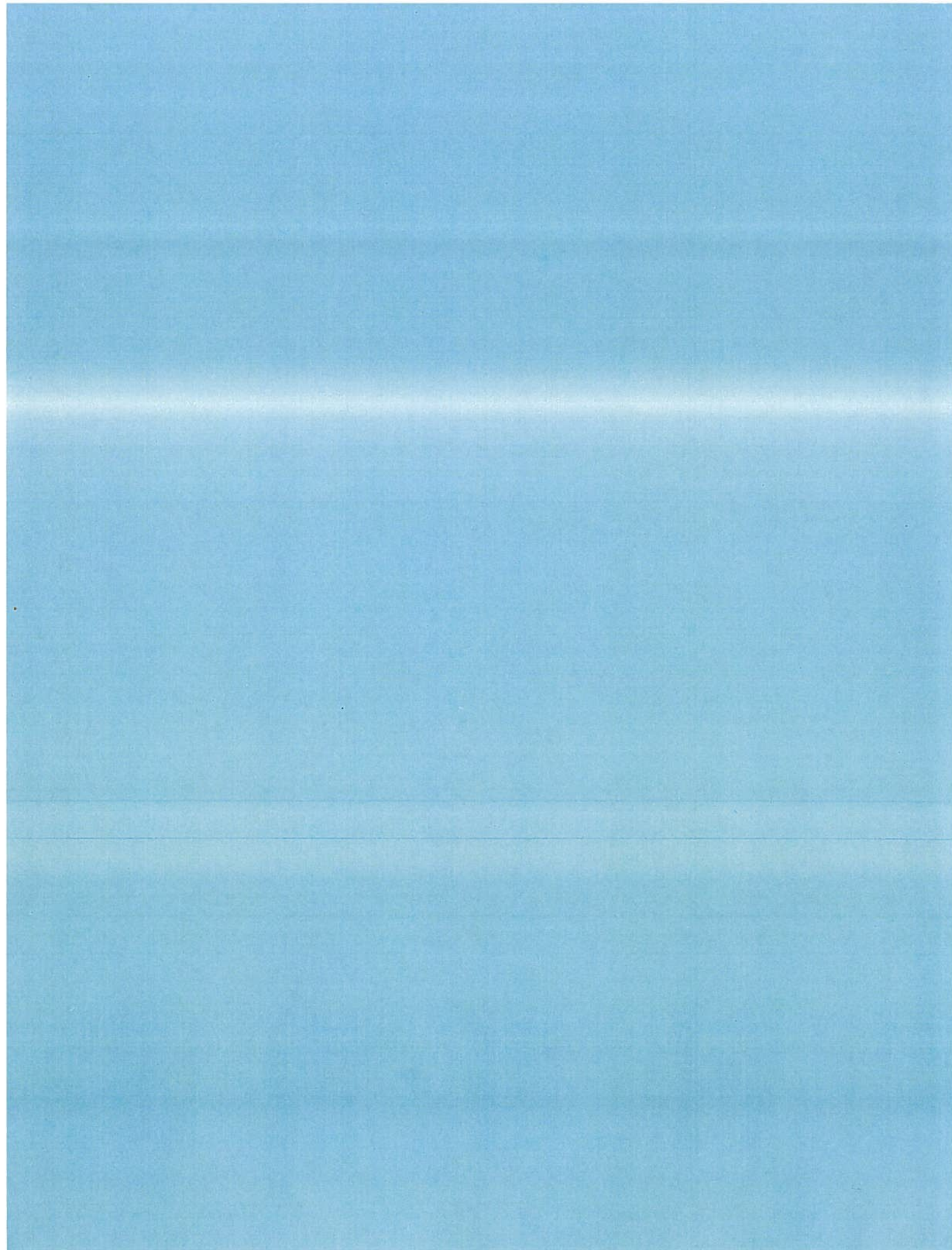


TABLE 1.1
SUMMARY OF SAMPLING AND ANALYSIS PROGRAM
WORK PLAN
GRAND RAPIDS METAL PLANT
WYOMING, MICHIGAN

<i>Investigation Activity</i>	<i>Sample Matrix</i>	<i>Field Parameters</i>	<i>Laboratory Parameters</i>	<i>Investigative Samples</i>	<i>Quality Control Samples (1)</i>			
					<i>Field Blanks (2)</i>	<i>Field Duplicates</i>	<i>MS/MSD (3)</i>	<i>Total</i>
Vertical Aquifer Sampling	Soil	PID Screen	TCL VOC	(4)	1/20	1/20	1/20	(5)
	Groundwater	pH/Temperature, DO, Specific Conductivity, Turbidity, ORP	TCL VOC	8	1	1	1	(5)
Monitoring Well Sampling	Groundwater	pH/Temperature, DO, Specific Conductivity, Turbidity, ORP	TCL VOC TCL SVOC (6)	40	2	2	2	(5)
LNAPL Characterization	LNAPL	None	TCL VOC, TCL SVOC, PCB TAL Metals, Total Cyanide, Hydrocarbon Fuel Scan, Average MW, Density, Dynamic Viscosity	1	0	0	0	1
Bulk Unload Area Evaluation	Soil	PID Screen	PCB, PNA, TAL metals	TBD	0	0	0	TBD
Waste Characterization	Waste	None	PCB, TCL VOC, TCLP VOC, TCLP SVOC, TCLP Metals, Corrosivity, Ignitability	(7)	0	0	0	(7)

Notes:

- (1) - A laboratory trip blank will be submitted with each shipment of samples for TCL VOC analysis.
- (2) - Field blank samples will not be required if dedicated or disposable sampling equipment is used.
- (3) - Matrix Spike/Matrix Spike duplicate (MS/MSD) analyses are required for samples submitted for organic and inorganic analyses are to be analyzed at a frequency of one per group of twenty (20) or fewer investigative samples for the activities detailed above.
- (4) - Collection of soil samples and laboratory analysis will be at the discretion of CRA's field personnel based on field conditions.
- (5) - The frequency of Field Blanks and Field Duplicates will be one per 20 investigative samples and will be combined from multiple investigation activities.
The total sample quantity will vary depending on which activity the QC samples are collected from.
- (6) - TCL SVOCs will be analyzed at specific locations.
- (7) - The quantity of waste samples will be based on volume of waste generated and quantity of drums.

PID = Photoionization Detector

DO = Dissolved Oxygen

ORP = Oxidation-Reduction Potential

LNAPL = Light Non-aqueous Phase Liquid

TCL = Target Compound List

TAL = Target Analyte List

VOC = Volatile Organic Compounds

SVOC = Semi-volatile Organic Compounds

PCB = Polychlorinated Biphenyls

PNA = Polynuclear Aromatics

TCLP = Toxicity Characteristic Leaching Procedure

MW = Molecular Weight

TBD = To Be Determined

TABLE 1.2

VERTICAL AQUIFER AND MONITORING WELL PARAMETER LIST
WORK PLAN
GRAND RAPIDS METAL PLANT
WYOMING, MICHIGAN

Compound	<i>Estimated</i>		<i>Method</i>	
	<i>Quantitation Limits (EQL) ¹</i>		<i>Detection Limits (MDL) ²</i>	
	<i>Water</i>	<i>Soil</i>	<i>Water</i>	<i>Soil</i>
	(µg/L)	(µg/kg)	(µg/L)	(µg/kg)
<i>Target Compound List (TCL) Volatile Organic Compounds (VOC) ³</i>				
Acetone	25	600	1.1	99
Benzene	1	40	0.13	6.4
Bromodichloromethane	1	80	0.15	12
Bromoform	1	80	0.64	13
Bromomethane	1	160	0.41	25
2-Butanone	25	600	0.57	49
Carbon disulfide	5	200	0.13	20
Carbon tetrachloride	1	40	0.13	12
Chlorobenzene	1	40	0.15	6.3
Chloroethane	1	200	0.18	6.9
Chloroform	1	40	0.29	65
Chloromethane	1	200	0.16	12
Cyclohexane	1	960	0.3	5.2
Dibromochloromethane	1	40	0.12	7.7
1,2-Dibromo-3-chloropropane	1	200	0.67	60
1,2-Dibromoethane	1	200	0.24	9.9
1,2-Dichlorobenzene	1	80	0.13	17
1,3-Dichlorobenzene	1	80	0.14	7.2
1,4-Dichlorobenzene	1	80	0.13	7.9
Dichlorodifluoromethane	1	80	0.31	5.4
1,1-Dichloroethane	1	125	0.15	7.3
1,2-Dichloroethane	1	40	0.22	9.3
1,1-Dichloroethene	1	40	0.17	14
cis-1,2-Dichloroethene	1	40	0.19	11
trans-1,2-Dichloroethene	1	40	0.19	8.3
1,2-Dichloropropane	1	40	0.18	7.4
cis-1,3-Dichloropropene	1	40	0.14	5.5
trans-1,3-Dichloropropene	1	40	0.19	5.4
Ethylbenzene	1	40	0.17	6.8
2-Hexanone	50	2,000	0.41	24
Isopropylbenzene	5	200	0.13	5.1
Methyl acetate	10	960	0.38	52
Methylene chloride	5	200	0.13	5.7
Methylcyclohexane	1	960	0.33	99
4-Methyl-2-pentanone	50	2,000	0.32	11
Methyl tert-butyl ether	5	200	0.17	10
Styrene	1	40	0.11	28
1,1,2,2-Tetrachloroethane	1	40	0.18	8.2
Tetrachloroethene	1	40	0.29	9

TABLE 1.2

VERTICAL AQUIFER AND MONITORING WELL PARAMETER LIST
WORK PLAN
GRAND RAPIDS METAL PLANT
WYOMING, MICHIGAN

Compound	Estimated Quantitation Limits (EQL) ¹		Method Detection Limits (MDL) ²	
	Water (µg/L)	Soil (µg/kg)	Water (µg/L)	Soil (µg/kg)
<i>TCL VOC (continued)</i>				
Toluene	1	40	0.13	9.1
1,2,4-Trichlorobenzene	5	200	0.15	12
1,1,1-Trichloroethane	1	40	0.22	9.5
1,1,2-Trichloroethane	1	40	0.27	9.9
Trichloroethene	1	40	0.17	12
Trichlorofluoromethane	1	80	0.21	8.3
1,1,2-Trichloro-1,2,2-trifluoroethane	1	200	0.28	10
Vinyl chloride	1	32	0.22	16
Xylenes (total)	3	120	0.28	15
<i>TCL Semi-Volatile Organic Compounds (SVOC) ³</i>				
Acenaphthene	5	264	0.054	1.3
Acenaphthylene	5	264	0.054	1.2
Acetophenone	5	264	0.55	9
Anthracene	5	264	0.054	1.3
Atrazine	5	40	0.65	21
Benzaldehyde	5	264	0.75	21
Benzo(a)anthracene	1	264	0.052	0.95
Benzo(a)pyrene	1	264	0.048	1.3
Benzo(b)fluoranthene	1	264	0.049	1.2
Benzo(g,h,i)perylene	1	264	0.053	1.3
Benzo(k)fluoranthene	1	264	0.049	1.7
1,1'-Biphenyl	5	264	0.55	23
4-Bromophenylphenyl ether	5	264	0.52	21
Butylbenzylphthalate	5	264	0.51	19
di-n-Butylphthalate	5	264	0.61	19
Caprolactam	10	264	0.61	37
Carbazole	10	264	0.54	19
4-Chloroaniline	10	264	0.56	17
bis(2-Chloroethoxy)methane	5	264	0.49	22
bis(2-Chloroethyl)ether	1	80	0.088	2
2,2'-oxibis(1-Chloropropane)	5	264	0.52	26
4-Chloro-3-methylphenol	5	264	0.41	21
2-Chloronaphthalene	5	264	0.62	22
2-Chlorophenol	5	264	0.039	26
4-Chlorophenyl phenyl ether	5	264	0.55	24
Chrysene	1	264	0.048	0.9
Dibenz(a,h)anthracene	2	264	0.039	1.3

TABLE 1.2

VERTICAL AQUIFER AND MONITORING WELL PARAMETER LIST
WORK PLAN
GRAND RAPIDS METAL PLANT
WYOMING, MICHIGAN

Compound	Targeted Quantitation Limits (EQL) ¹		Method Detection Limits (MDL) ²	
	Water (µg/L)	Soil (µg/kg)	Water (µg/L)	Soil (µg/kg)
<i>TCL Semi-Volatile Organic Compounds (cont'd)</i>				
Dibenzofuran	4	264	0.54	20
3,3'-Dichlorobenzidine	4	1600	0.48	18
2,4-Dichlorophenol	10	264	1.1	20
Diethylphthalate	5	264	0.63	19
2,4-Dimethylphenol	5	264	0.56	20
Dimethylphthalate	5	264	0.44	21
4,6-Dinitro-2-methylphenol	20	150	0.27	13
2,4-Dinitrophenol	20	330	3.5	83
2,4-Dinitrotoluene	5	264	0.4	18
2,6-Dinitrotoluene	5	264	0.47	21
bis(2-Ethylhexyl)phthalate	5	264	0.88	18
Fluoranthene	1	264	0.036	1.2
Fluorene	5	264	0.043	1.2
Hexachlorobenzene	1	264	0.065	2.1
Hexachlorobutadiene	5	40	0.51	26
Hexachlorocyclopentadiene	5	264	0.74	16
Hexachloroethane	5	264	0.58	28
Indeno(1,2,3-cd)pyrene	2	264	0.065	1.5
Isophorone	5	264	0.5	21
2-Methylnaphthalene	5	264	0.061	1.5
2-Methylphenol	5	264	0.56	28
4-Methylphenol	5	264	0.64	22
Naphthalene	5	264	0.069	1.6
2-Nitroaniline	20	200	0.43	22
3-Nitroaniline	20	200	0.67	16
4-Nitroaniline	20	200	0.47	26
Nitrobenzene	3	264	0.053	2.2
2-Nitrophenol	5	264	1.3	19
4-Nitrophenol	20	330	0.63	110
N-Nitroso-di-n-propylamine	5	264	0.53	23
N-Nitrosodiphenylamine (diphenylamine)	5	264	0.46	21
di-n-Octylphthalate	5	264	0.39	18
Pentachlorophenol	20	150	0.48	82
Phenanthrene	2	264	0.087	2
Phenol	5	264	0.96	25
Pyrene	5	264	0.048	1.1
2,4,5-Trichlorophenol	5	264	0.96	25
2,4,6-Trichlorophenol	4	264	1.4	21

TABLE 1.2

VERTICAL AQUIFER AND MONITORING WELL PARAMETER LIST
WORK PLAN
GRAND RAPIDS METAL PLANT
WYOMING, MICHIGAN

Notes:

- ¹ - Please note that these are targeted quantitation limits and 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.
- ² - Method Detection Limits (MDL) are also presented for guidance only. Actual MDLs will vary depending on sample specific preparation factors. The MDLs are also highly matrix dependant and may be elevated due to matrix effects, QA/QC problems and high concentrations of target and non-target analytes. Laboratory MDLs are updated on a periodic basis and the MDLs in effect when the samples are analyzed will be used for reporting purposes.
- ³ - U.S. EPA Contract Laboratory Program, "Statement of Work for Organic Analysis, Multi-Media, Multi-Concentration", OLM4.2, May 1999 Target Compound List.

TABLE 1.3

LNAPL PARAMETER LIST
WORK PLAN
GRAND RAPIDS METAL PLANT
WYOMING, MICHIGAN

Compound	Estimated	Method
	Quantitation Limits (TQL) ¹ (µg/kg)	Detection Limits (MDL) ² (µg/kg)
<i>Target Compound List (TCL) Volatile Organic Compounds (VOC) ³</i>		
Acetone	2,500	99
Benzene	620	6.4
Bromodichloromethane	620	12
Bromoform	620	13
Bromomethane	1,200	25
2-Butanone	2,500	49
Carbon disulfide	620	20
Carbon tetrachloride	620	12
Chlorobenzene	620	6.3
Chloroethane	1,200	6.9
Chloroform	620	65
Chloromethane	1,200	12
Cyclohexane	2,500	5.2
Dibromochloromethane	620	7.7
1,2-Dibromo-3-chloropropane	1,200	60
1,2-Dibromoethane	620	9.9
1,2-Dichlorobenzene	1,200	17
1,3-Dichlorobenzene	1,200	7.2
1,4-Dichlorobenzene	1,200	7.9
Dichlorodifluoromethane	1,200	5.4
1,1-Dichloroethane	620	7.3
1,2-Dichloroethane	620	9.3
1,1-Dichloroethene	620	14
cis-1,2-Dichloroethene	310	11
trans-1,2-Dichloroethene	310	8.3
1,2-Dichloropropane	620	7.4
cis-1,3-Dichloropropene	620	5.5
trans-1,3-Dichloropropene	620	5.4
Ethylbenzene	620	6.8
2-Hexanone	2,500	24
Isopropylbenzene	1,200	5.1
Methyl acetate	1,200	52
Methylene chloride	620	8.92
Methylcyclohexane	620	99
4-Methyl-2-pentanone	2,500	11
Methyl tert-butyl ether	2,500	10
Styrene	620	28
1,1,2,2-Tetrachloroethane	620	8.2
Tetrachloroethene	620	9
Toluene	620	9.1

TABLE 1.3

LNAPL PARAMETER LIST
WORK PLAN
GRAND RAPIDS METAL PLANT
WYOMING, MICHIGAN

Compound	Estimated	Method
	Quantitation Limits (TQL) ¹ (µg/kg)	Detection Limits (MDL) ² (µg/kg)
<i>TCL VOC (continued)</i>		
1,2,4-Trichlorobenzene	620	12
1,1,1-Trichloroethane	620	9.5
1,1,2-Trichloroethane	1,200	9.9
Trichloroethene	620	12
Trichlorofluoromethane	1,200	8.3
1,1,2-Trichloro-1,2,2-trifluoroethane	2,500	10
Vinyl chloride	1,200	16
Xylenes (total)	620	15
<i>TCL Semi-Volatile Organic Compounds (SVOC)</i>		
Acenaphthene	20,000	55
Acenaphthylene	20,000	84
Acetophenone	20,000	330
Anthracene	20,000	138
Atrazine	20,000	660
Benzaldehyde	20,000	414
Benzo(a)anthracene	20,000	84
Benzo(a)pyrene	20,000	138
Benzo(b)fluoranthene	20,000	138
Benzo(g,h,i)perylene	20,000	108
Benzo(k)fluoranthene	20,000	138
1,1'-Biphenyl	20,000	432
4-Bromophenylphenyl ether	20,000	282
Butylbenzylphthalate	20,000	222
di-n-Butylphthalate	20,000	300
Caprolactam	20,000	468
Carbazole	20,000	558
4-Chloroaniline	20,000	312
bis(2-Chloroethoxy)methane	20,000	960
bis(2-Chloroethyl)ether	20,000	246
2,2'-oxibis(1-Chloropropane)	20,000	312
4-Chloro-3-methylphenol	20,000	3,060
2-Chloronaphthalene	20,000	378
2-Chlorophenol	20,000	216
4-Chlorophenyl phenyl ether	20,000	216
Chrysene	20,000	59
Dibenz(a,h)anthracene	20,000	90
Dibenzofuran	20,000	50
3,3'-Dichlorobenzidine	96,000	294

TABLE 1.3

LNAPL PARAMETER LIST
WORK PLAN
GRAND RAPIDS METAL PLANT
WYOMING, MICHIGAN

<i>Compound</i>	<i>Estimated</i>	<i>Method</i>
	<i>Quantitation Limits (TQL) ¹</i>	<i>Detection Limits (MDL) ²</i>
	<i>(µg/kg)</i>	<i>(µg/kg)</i>
<i>TCL Semi-Volatile Organic Compounds (cont'd)</i>		
2,4-Dichlorophenol	20,000	318
Diethylphthalate	20,000	372
2,4-Dimethylphenol	20,000	408
Dimethylphthalate	20,000	384
4,6-Dinitro-2-methylphenol	96,000	2,820
2,4-Dinitrophenol	96,000	2,460
2,4-Dinitrotoluene	20,000	342
2,6-Dinitrotoluene	20,000	348
bis(2-Ethylhexyl)phthalate	20,000	960
Fluoranthene	20,000	56
Fluorene	20,000	78
Hexachlorobenzene	20,000	84
Hexachlorobutadiene	20,000	150
Hexachlorocyclopentadiene	96,000	174
Hexachloroethane	20,000	294
Indeno(1,2,3-cd)pyrene	20,000	120
Isophorone	20,000	210
2-Methylnaphthalene	20,000	59
2-Methylphenol	20,000	402
4-Methylphenol	20,000	348
Naphthalene	20,000	53
2-Nitroaniline	96,000	306
3-Nitroaniline	96,000	192
4-Nitroaniline	96,000	216
Nitrobenzene	20,000	384
2-Nitrophenol	20,000	204
4-Nitrophenol	96,000	4,860
N-Nitroso-di-n-propylamine	20,000	456
N-Nitrosodiphenylamine (diphenylamine)	20,000	246
di-n-Octylphthalate	20,000	660
Pentachlorophenol	20,000	2,700
Phenanthrene	20,000	66
Phenol	20,000	342
Pyrene	20,000	60
2,4,5-Trichlorophenol	20,000	294
2,4,6-Trichlorophenol	20,000	408

TABLE 1.3

LNAPL PARAMETER LIST
WORK PLAN
GRAND RAPIDS METAL PLANT
WYOMING, MICHIGAN

<i>Compound</i>	<i>Estimated Quantitation Limits (TQL)¹ (µg/kg)</i>	<i>Method Detection Limits (MDL)² (µg/kg)</i>
<i>Polychlorinated Biphenyls (PCB) as Aroclors</i>		
Aroclor-1016	1000	190
Aroclor-1221	1000	220
Aroclor-1232	1000	170
Aroclor-1242	1000	290
Aroclor-1248	1000	200
Aroclor-1254	1000	120
Aroclor-1260	1000	130
<i>Compound</i>	<i>Estimated Quantitation Limits (TQL)¹ (mg/kg)</i>	<i>Method Detection Limits (MDL)² (mg/kg)</i>
<i>TAL Metals</i>		
Aluminum	20	9.6
Antimony	6	0.39
Arsenic	30	0.3
Barium	20	0.071
Beryllium	0.5	0.043
Cadmium	0.5	0.036
Chromium	1	0.2
Cobalt	5	0.16
Copper	2.5	0.74
Iron	10	4.9
Lead	10	0.19
Manganese	1.5	0.074
Mercury	0.1	0.015
Nickel	4	0.27
Selenium	25	0.45
Silver	1	0.1
Thallium	200	0.55
Vanadium	5	0.12
Zinc	2	1

TABLE 1.3

**LNAPL PARAMETER LIST
WORK PLAN
GRAND RAPIDS METAL PLANT
WYOMING, MICHIGAN**

Notes:

- ¹ - Please note that these are targeted quantitation limits and 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.
- ² - Method Detection Limits (MDL) are also presented for guidance only. Actual MDLs will vary depending on sample specific preparation factors. The MDLs are also highly matrix dependant and may be elevated due to matrix effects, QA/QC problems and high concentrations of target and non-target analytes. Laboratory MDLs are updated on a periodic basis and the MDLs in effect when the samples are analyzed will be used for reporting purposes.
- ³ - U.S. EPA Contract Laboratory Program, "Statement of Work for Organic Analysis, Multi-Media, Multi-Concentration", OLM4.2, May 1999 Target Compound List.

TABLE 1.4

SOIL SAMPLE PARAMETER LIST
WORK PLAN
GRAND RAPIDS METAL PLANT
WYOMING, MICHIGAN

Compound	Estimated	Method
	Quantitation Limits (EQL) ¹ (µg/kg)	Detection Limits (MDL) ² (µg/kg)
<i>Target Compound List (TCL) Volatile Organic Compounds (VOC) ³</i>		
Acetone	600	99
Benzene	40	6.4
Bromodichloromethane	80	12
Bromoform	80	13
Bromomethane	160	25
2-Butanone	600	49
Carbon disulfide	200	20
Carbon tetrachloride	40	12
Chlorobenzene	40	6.3
Chloroethane	200	6.9
Chloroform	40	65
Chloromethane	200	12
Cyclohexane	960	5.2
Dibromochloromethane	40	7.7
1,2-Dibromo-3-chloropropane	200	60
1,2-Dibromoethane	200	9.9
1,2-Dichlorobenzene	80	17
1,3-Dichlorobenzene	80	7.2
1,4-Dichlorobenzene	80	7.9
Dichlorodifluoromethane	80	5.4
1,1-Dichloroethane	125	7.3
1,2-Dichloroethane	40	9.3
1,1-Dichloroethene	40	14
cis-1,2-Dichloroethene	40	11
trans-1,2-Dichloroethene	40	8.3
1,2-Dichloropropane	40	7.4
cis-1,3-Dichloropropene	40	5.5
trans-1,3-Dichloropropene	40	5.4
Ethylbenzene	40	6.8
2-Hexanone	2,000	24
Isopropylbenzene	200	5.1
Methyl acetate	960	52
Methylene chloride	200	5.7
Methylcyclohexane	960	99
4-Methyl-2-pentanone	2,000	11
Methyl tert-butyl ether	200	10
Styrene	40	28
1,1,2,2-Tetrachloroethane	40	8.2
Tetrachloroethene	40	9

TABLE 1.4

SOIL SAMPLE PARAMETER LIST
WORK PLAN
GRAND RAPIDS METAL PLANT
WYOMING, MICHIGAN

Compound	Estimated	Method
	Quantitation Limits (EQL) ¹ (µg/kg)	Detection Limits (MDL) ² (µg/kg)
<i>TCL VOC (continued)</i>		
Toluene	40	9.1
1,2,4-Trichlorobenzene	200	12
1,1,1-Trichloroethane	40	9.5
1,1,2-Trichloroethane	40	9.9
Trichloroethene	40	12
Trichlorofluoromethane	80	8.3
1,1,2-Trichloro-1,2,2-trifluoroethane	200	10
Vinyl chloride	32	16
Xylenes (total)	120	15
<i>Polychlorinated Biphenyls (PCB) as Aroclors</i>		
Aroclor-1016	264	21
Aroclor-1221	264	16
Aroclor-1232	264	14
Aroclor-1242	264	13
Aroclor-1248	264	17
Aroclor-1254	264	17
Aroclor-1260	264	17
<i>Polynuclear Aromatic Hydrocarbons (PNA)</i>		
Acenaphthene	264	1.3
Acenaphthylene	264	1.2
Anthracene	264	1.3
Benzo(a)anthracene	264	0.95
Benzo(a)pyrene	264	1.3
Benzo(b)fluoranthene	264	1.2
Benzo(g,h,i)perylene	264	1.3
Benzo(k)fluoranthene	264	1.7
Chrysene	264	0.9
Dibenzo(a,h)anthracene	264	1.3
Fluoranthene	264	1.2
Fluorene	264	1.2
Indeno(1,2,3-cd)pyrene	264	1.5
2-Methylnaphthalene	264	1.5
Naphthalene	264	1.6
Phenanthrene	264	2
Pyrene	264	1.1

TABLE 1.4

SOIL SAMPLE PARAMETER LIST
WORK PLAN
GRAND RAPIDS METAL PLANT
WYOMING, MICHIGAN

<i>Compound</i>	<i>Estimated</i>	<i>Method</i>
	<i>Quantitation Limits (EQL) ¹</i>	<i>Detection Limits (MDL) ²</i>
	<i>(µg/kg)</i>	<i>(µg/kg)</i>
<i>TAL Inorganics</i>		
Aluminum	20,000	9600
Antimony	240	24
Arsenic	80	62
Barium	800	46
Beryllium	400	47
Cadmium	160	7.8
Calcium	400,000	16000
Chromium Total	1,600	200
Cobalt	400	3.8
Copper	800	43
Iron	10,000	4900
Lead	800	190
Magnesium	100,000	5100
Manganese	800	74
Mercury	40	15
Nickel	800	21
Potassium	500,000	6200
Selenium	160	90
Silver	80	16
Sodium	100,000	2400
Thallium	400	13
Vanadium	800	41
Zinc	1,000	440
Lead-Fine Fraction (6010B)	800	190
Lead Coarse Fraction (6010B)	800	190

Notes:

- ¹ - Please note that these are targeted quantitation limits and 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.
- ² - Method Detection Limits (MDL) are also presented for guidance only. Actual MDLs will vary depending on sample specific preparation factors. The MDLs are also highly matrix dependant and may be elevated due to matrix effects, QA/QC problems and high concentrations of target and non-target analytes. Laboratory MDLs are updated on a periodic basis and the MDLs in effect when the samples are analyzed will be used for reporting purposes.

TABLE 1.5
WASTE CHARACTERIZATION PARAMETER LIST
WORK PLAN
GRAND RAPIDS METAL PLANT
WYOMING, MICHIGAN

Compound	Estimated Quantitation Limits (TQL) ¹		Method Detection Limits (MDL) ²	
	Water (µg/L)	Soil (µg/kg)	Water (µg/L)	Soil (µg/kg)
<i>Target Compound List (TCL) Volatile Organic Compounds (VOC) ³</i>				
Acetone	25	600	1.1	99
Benzene	1	40	0.13	6.4
Bromodichloromethane	1	80	0.15	12
Bromoform	1	80	0.64	13
Bromomethane	1	160	0.41	25
2-Butanone	25	600	0.57	49
Carbon disulfide	5	200	0.13	20
Carbon tetrachloride	1	40	0.13	12
Chlorobenzene	1	40	0.15	6.3
Chloroethane	1	200	0.18	6.9
Chloroform	1	40	0.29	65
Chloromethane	1	200	0.16	12
Cyclohexane	1	960	0.3	5.2
Dibromochloromethane	1	40	0.12	7.7
1,2-Dibromo-3-chloropropane	1	200	0.67	60
1,2-Dibromoethane	1	200	0.24	9.9
1,2-Dichlorobenzene	1	80	0.13	17
1,3-Dichlorobenzene	1	80	0.14	7.2
1,4-Dichlorobenzene	1	80	0.13	7.9
Dichlorodifluoromethane	1	80	0.31	5.4
1,1-Dichloroethane	1	125	0.15	7.3
1,2-Dichloroethane	1	40	0.22	9.3
1,1-Dichloroethene	1	40	0.17	14
cis-1,2-Dichloroethene	1	40	0.19	11
trans-1,2-Dichloroethene	1	40	0.19	8.3
1,2-Dichloropropane	1	40	0.18	7.4
cis-1,3-Dichloropropene	1	40	0.14	5.5
trans-1,3-Dichloropropene	1	40	0.19	5.4
Ethylbenzene	1	40	0.17	6.8
2-Hexanone	50	2,000	0.41	24
Isopropylbenzene	5	200	0.13	5.1
Methyl acetate	10	960	0.38	52
Methylene chloride	5	200	0.13	5.7
Methylcyclohexane	1	960	0.33	99
4-Methyl-2-pentanone	50	2,000	0.32	11
Methyl tert-butyl ether	5	200	0.17	10
Styrene	1	40	0.11	28
1,1,2,2-Tetrachloroethane	1	40	0.18	8.2
Tetrachloroethene	1	40	0.29	9

TABLE 1.5

WASTE CHARACTERIZATION PARAMETER LIST
WORK PLAN
GRAND RAPIDS METAL PLANT
WYOMING, MICHIGAN

<i>Compound</i>	<i>Estimated Quantitation Limits (TQL) ¹</i>		<i>Method Detection Limits (MDL) ²</i>	
	<i>Water (µg/L)</i>	<i>Soil (µg/kg)</i>	<i>Water (µg/L)</i>	<i>Soil (µg/kg)</i>
<i>TCL VOC (continued)</i>				
Toluene	1	40	0.13	9.1
1,2,4-Trichlorobenzene	5	200	0.15	12
1,1,1-Trichloroethane	1	40	0.22	9.5
1,1,2-Trichloroethane	1	40	0.27	9.9
Trichloroethene	1	40	0.17	12
Trichlorofluoromethane	1	80	0.21	8.3
1,1,2-Trichloro-1,2,2-trifluoroethane	1	200	0.28	10
Vinyl chloride	1	32	0.22	16
Xylenes (total)	3	120	0.28	15
<i>Polychlorinated Biphenyls (PCB) as Aroclors</i>				
Aroclor-1016	0.2	264	0.044	21
Aroclor-1221	0.2	264	0.045	16
Aroclor-1232	0.2	264	0.073	14
Aroclor-1242	0.2	264	0.06	13
Aroclor-1248	0.2	264	0.061	17
Aroclor-1254	0.2	264	0.032	17
Aroclor-1260	0.2	264	0.038	17
<i>Compound</i>	<i>Estimated Quantitation Limits (TQL) ¹</i>		<i>Method Detection Limits (MDL) ²</i>	
	<i>Waste Leachate (mg/L)</i>		<i>Waste Leachate (mg/L)</i>	
<i>Toxic Characteristic Leachate Procedure (TCLP) VOC</i>				
Benzene	0.025		0.00013	
2-Butanone	0.050		0.00057	
Carbon tetrachloride	0.025		0.00013	
Chlorobenzene	0.025		0.00015	
Chloroform	0.025		0.00016	
1,2-Dichloroethane	0.025		0.00022	
1,1-Dichloroethene	0.07		0.00019	
Tetrachloroethene	0.07		0.00029	
Trichloroethene	0.05		0.00017	
Vinyl chloride	0.025		0.00022	

TABLE 1.5

WASTE CHARACTERIZATION PARAMETER LIST
WORK PLAN
GRAND RAPIDS METAL PLANT
WYOMING, MICHIGAN

<i>Compound</i>	<i>Estimated</i>	<i>Method</i>
	<i>Quantitation Limits (TQL) ¹</i>	<i>Detection Limits (MDL) ²</i>
	<i>Waste Leachate</i>	<i>Waste Leachate</i>
	<i>(mg/L)</i>	<i>(mg/L)</i>
TCLP SVOC		
Cresols, total	0.15	0.00075
1,4-Dichlorobenzene	0.05	0.00052
2,4-Dinitrotoluene	0.05	0.0004
Hexachlorobenzene	0.05	0.000065
Hexachlorobutadiene	0.05	0.00051
Hexachloroethane	0.05	0.00058
Nitrobenzene	0.05	0.000053
Pentachlorophenol	0.1	0.00048
Pyridine	0.1	0.00078
2,4,5-Trichlorophenol	0.25	0.00096
2,4,6-Trichlorophenol	0.05	0.0014
TCLP Metals		
Arsenic	0.5	0.0032
Barium	10	0.00067
Cadmium	0.1	0.00066
Chromium	0.5	0.0022
Lead	0.5	0.0019
Mercury	0.002	0.00012
Selenium	0.25	0.0041
Silver	0.5	0.0022
	<i>Estimated</i>	<i>Method</i>
	<i>Quantitation Limits (TQL) ¹</i>	<i>Detection Limits (MDL) ²</i>
	<i>Waste</i>	<i>Waste</i>
	<i>(mg/kg)</i>	<i>(mg/kg)</i>
Waste Characteristics		
Ignitability (flashpoint)	---	NA
Corrosivity (pH)	---	NA

TABLE 1.5

**WASTE CHARACTERIZATION PARAMETER LIST
WORK PLAN
GRAND RAPIDS METAL PLANT
WYOMING, MICHIGAN**

Notes:

- ¹ - Please note that these are targeted quantitation limits and 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.
- ² - Method Detection Limits (MDL) are also presented for guidance only. Actual MDLs will vary depending on sample specific preparation factors. The MDLs are also highly matrix dependant and may be elevated due to matrix effects, QA/QC problems and high concentrations of target and non-target analytes. Laboratory MDLs are updated on a periodic basis and the MDLs in effect when the samples are analyzed will be used for reporting purposes.
- ³ - U.S. EPA Contract Laboratory Program, "Statement of Work for Organic Analysis, Multi-Media, Multi-Concentration", OLM4.2, May 1999 Target Compound List.

TABLE 4.1
CONTAINER, PRESERVATION, SHIPPING AND PACKAGING REQUIREMENTS
WORK PLAN
GRAND RAPIDS METAL PLANT
WYOMING, MICHIGAN

<i>Analyses</i>	<i>Sample Containers¹</i>	<i>Preservation</i>	<i>Maximum Holding Time from Sample Collection²</i>	<i>Volume of Sample</i>	<i>Shipping</i>	<i>Normal Packaging</i>
WATER (Groundwater)						
Volatile Organic Compounds (VOC)	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-wrap
Semi-volatile Organic Compounds (SVOC)	Two 1 liter amber 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	Foam Liner or Bubble-wrap
SOLID (Soil)						
TCL VOC - Low Level	Two 5g aliquots per analysis (4) One 10 gram aliquot per analysis (4)	5 milliter purified water 10 milliliters methanol Iced, 4 ± 2° C	48 hours until frozen 14 days for analysis 14 days for analysis	Fill completely	Overnight or Hand Deliver	Foam Liner or Bubble-wrap
SVOC	One 4-ounce glass jar ⁴	Iced, 4 ± 2° C	14 days for SVOC extraction 40 days after extraction for analysis	Fill to shoulder of jar	Overnight or Hand Deliver	Foam Liner or Bubble-wrap
Polychlorinated Biphenyls (PCB)	One 4-ounce glass jar ⁴	Iced, 4 ± 2° C	14 days for extraction 40 days after extraction for analysis	Fill to shoulder of jar	Overnight or Hand Deliver	Foam Liner or Bubble-wrap

TABLE 4.1
CONTAINER, PRESERVATION, SHIPPING AND PACKAGING REQUIREMENTS
WORK PLAN
GRAND RAPIDS METAL PLANT
WYOMING, MICHIGAN

<i>Analyses</i>	<i>Sample Containers¹</i>	<i>Preservation</i>	<i>Maximum Holding Time from Sample Collection²</i>	<i>Volume of Sample</i>	<i>Shipping</i>	<i>Normal Packaging</i>
<i>SOLID (Soil), continued</i>						
Polynuclear Aromatics (PNA)	One 4-ounce glass jar ⁴	Iced, 4 ± 2° C	14 days for extraction 40 days after extraction for analysis	Fill to shoulder of jar	Overnight or Hand Deliver	Foam Liner or Bubble-wrap
Metals	One 4-ounce glass jar ⁴	Iced, 4 ± 2° C	180 days (mercury 28 days) for analysis	Fill to shoulder of jar	Overnight or Hand Deliver	Foam Liner or Bubble-wrap
<i>Light Non-Aqueous Phase Liquid (LNAPL)</i>						
VOC	One 4-ounce glass jar ⁴	Iced, 4 ± 2° C	14 days for analysis	Fill completely	Overnight or Hand Deliver	Foam Liner or Bubble-wrap
SVOC	One 4-ounce glass jar ⁴	Iced, 4 ± 2° C	14 days for extraction 40 days after extraction for analysis	Fill to shoulder of jar	Overnight or Hand Deliver	Foam Liner or Bubble-wrap
PCB	One 4-ounce glass jar ⁴	Iced, 4 ± 2° C	14 days for extraction 40 days after extraction for analysis	Fill to shoulder of jar	Overnight or Hand Deliver	Foam Liner or Bubble-wrap
Metals	One 4-ounce glass jar ⁴	Iced, 4 ± 2° C	180 days (mercury 28 days) for analysis	Fill to shoulder of jar	Overnight or Hand Deliver	Foam Liner or Bubble-wrap

TABLE 4.1

CONTAINER, PRESERVATION, SHIPPING AND PACKAGING REQUIREMENTS
WORK PLAN
GRAND RAPIDS METAL PLANT
WYOMING, MICHIGAN

<i>Analyses</i>	<i>Sample Containers¹</i>	<i>Preservation</i>	<i>Maximum Holding Time from Sample Collection²</i>	<i>Volume of Sample</i>	<i>Shipping</i>	<i>Normal Packaging</i>
<i>LNAPL, continued</i>						
Cyanide	One 4-ounce glass jar ⁴	Iced, 4 ± 2° C	14 days for analysis	Fill to shoulder of jar	Overnight or Hand Deliver	Foam Liner or Bubble-wrap
Density, Viscosity	One 4-ounce glass jar ⁴	Iced, 4 ± 2° C	180 days for analysis	Fill to shoulder of jar	Overnight or Hand Deliver	Foam Liner or Bubble-wrap
<i>Waste (Solid)</i>						
VOC (totals)	One 4-ounce glass jar ⁴	Iced, 4 ± 2° C	14 days for analysis	Fill completely	Overnight or Hand Deliver	Foam Liner or Bubble-wrap
VOC (TCLP)	One 4-ounce glass jar ⁴	Iced, 4 ± 2° C	14 days for extraction 14 days for analysis	Fill completely	Overnight or Hand Deliver	Foam Liner or Bubble-wrap
<i>Waste (Solid), continued</i>						
SVOC (TCLP)	One 4-ounce glass jar ⁴	Iced, 4 ± 2° C	14 days for TCLP extraction 14 days for SVOC extraction 40 days after extraction for analysis	Fill to shoulder of jar	Overnight or Hand Deliver	Foam Liner or Bubble-wrap
Metals (TCLP)	One 4-ounce glass jar ⁴	Iced, 4 ± 2° C	180 days (mercury 28 days) for extraction 180 days (mercury 28 days) for analysis	Fill to shoulder of jar	Overnight or Hand Deliver	Foam Liner or Bubble-wrap
Corrosivity (pH) and Ignitability (flash point)	One 4-ounce glass jar ⁴	Iced, 4 ± 2° C	As soon as practical	Fill to should of jar	Overnight or Hand Deliver	Foam Liner or Bubble-wrap

TABLE 4.1
CONTAINER, PRESERVATION, SHIPPING AND PACKAGING REQUIREMENTS
WORK PLAN
GRAND RAPIDS METAL PLANT
WYOMING, MICHIGAN

<i>Analyses</i>	<i>Sample Containers¹</i>	<i>Preservation</i>	<i>Maximum Holding Time from Sample Collection²</i>	<i>Volume of Sample</i>	<i>Shipping</i>	<i>Normal Packaging</i>
<i>Waste (Liquid)</i>						
VOC (totals)	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-wrap
VOC (TCLP)	Two 40 mL teflon-lined septum vials	Iced, 4 ± 2° C	7 days for extraction 14 days for analysis	Fill completely, no air bubbles	Overnight or Hand Deliver	Foam Liner or Bubble-wrap
<i>Waste (Liquid), continued</i>						
SVOC (TCLP)	One 1 liter amber glass bottle per analysis ⁴	Iced, 4 ± 2° C	7 days for TCLP extraction 7 days for SVOC extraction 40 days after extraction for analysis	Fill to neck of bottle	Overnight or Hand Deliver	Foam Liner or Bubble-wrap
Metals (TCLP)	One 1 liter amber glass bottle per analysis ⁴	Iced, 4 ± 2° C	180 days (mercury 28 days) for extraction 180 days (mercury 28 days) for analysis	Fill to neck of bottle	Overnight or Hand Deliver	Foam Liner or Bubble-wrap
Corrosivity (pH) and Ignitability (flash point)	One 1 liter amber glass bottle ⁴	Iced, 4 ± 2° C	As soon as practical	Fill to neck of bottle	Overnight or Hand Deliver	Foam Liner or Bubble-wrap

Notes:

¹ - Multiple parameters on a single sample may be combined into one single 16 ounce glass jar.

² - These are technical holding times, i.e., are based on time elapsed from time of sample collection.

³ - Soil VOC samples will require in addition to the EnCore an additional container of sample for dry weight determination if no other analyses are required. Sediment samples if non-cohesive, may require standard bulk sampling techniques using a 4 ounce glass jar with no head space.

⁴ - The NAPL and Waste Characterization samples may be combined into single containers of different volume depending on the parameters required and the material sampled.

TABLE 7.1

SUMMARY OF ANALYTICAL METHODS
WORK PLAN
GRAND RAPIDS METAL PLANT
WYOMING, MICHIGAN

<i>Parameter</i> ¹	<i>Preparation Method</i> ²	<i>Laboratory Preparation SOP</i>	<i>Analytical Method</i> ²	<i>Laboratory Analytical SOP</i>
<u>Groundwater Samples</u>				
VOC	SW-846 5030B	NC-MS-019	SW-846 8260B	NC-MS-019
SVOC	SW-846 3510C	NC-OP-032	SW-846 8270C	NC-GC-038
<u>Soil Samples</u>				
VOC	SW-846 5035 ⁴	NC-MS-019	SW-846 8260B	NC-MS-019
PNA	SW-846 3550B	NC-OP-032	SW-846 8270C	NC-GC-038
PCB	SW-846 3545	NC-OP-032	SW-846 8082	NC-GC-038
Metals				
ICP Metals	SW-846 3050A	NC-IP-010	SW-846 6010B	NC-MT-012
ICP/MS Metals	SW-846 3050A	NC-IP-010	SW-846 6020	NC-MT-0002
Mercury	SW-846 7471A	NC-MT-0011	SW-846 7471A	NC-MT-0011
<u>LNAFL Samples</u>				
VOC	SW-846 5030B	NC-MS-019	SW-846 8260B	NC-MS-019
SVOC	SW-846 3550B	NC-OP-032	SW-846 8270C	NC-GC-038
PCB	SW-846 3550B	NC-OP-0032	SW-846 8082	NC-GC-038
Metals				
ICP Metals	SW-846 3050A	NC-IP-010	SW-846 6010B	NC-MT-012
ICP/MS Metals	SW-846 3050A	NC-IP-010	SW-846 6020	NC-MT-0002
Mercury	SW-846 7471A	NC-MT-011	SW-846 7471A	NC-MT-011
Cyanide	SW-846 9012A	NC-WC-0032	SW-8469012A	NC-WC-0031
Hydrocarbon Fuel Scan	SW-846 8015B	SW-846 8015B	SW-846 8015B	SW-846 8015B
Average MW	NA	NA	Calculation	Calculation
Density	ASTM D 1298	PR-0Q-007 Rev1	ASTM D 1298	PR-0Q-007 Rev1
Dynamic Viscosity	ASTM D 445	PR-0Q-008 Rev1	ASTM D 445	PR-0Q-008 Rev1
<u>Waste Characterization Samples</u>				
PCB (solids)	SW-846 3500C/3550B	NC-OP-032	SW-846 8082	NC-GC-038
PCB (waters)	SW-846 3500C	NC-OP-032	SW-846 8082	NC-GC-038
TCLP Extraction	SW-846 1311	NC-OP-033	NA	NA
TCLP VOC	SW-846 5030B/5035 ⁴	NC-MS-019	SW-846 8260B	NC-MS-019
TCLP SVOC	SW-846 3500B/3550B	NC-OP-032	SW-846 8270C	NC-GC-038
TCLP Metals				
ICP Metals	SW-846 3010A	NC-IP-010	SW-846 6010B	NC-MT-012
Mercury	SW-846 7470A	NC-MT-011	SW-846 7470A	NC-MT-011
Ignitability (flashpoint)	NA	NA	SW-846 1010	NC-WC-0034
Corrosivity (pH)	SW-846 9045C	NC-WC-010	SW-846 9045C	NC-WC-010

Notes:

¹ Refer to Table 1.2 for the compounds/elements of each parameter group.

² Preparation and Analytical Method References:

- SW-846 - "Test Methods for Evaluating Solid Wastes, Physical/Chemical Methods", SW-846, 3rd Edition, and Promulgated Updates, November 1986.
- ASTM - "Annual Book of ASTM Standards",

³ Metals analysis includes ICP and ICP/MS for TAL Metals as follows:

- ICP Metals - Al, Ca, Cr, Fe, Pb, Mg, Mn, K, Na
- ICP/MS Metals include - Sb, As, Ba, Be, Cd, Co, Cu, Ni, Se, Ag, Tl, V, Zn.

⁴ Sediment and sludge samples may be collected as bulk samples which will be prepared in accordance with SW-846 Method 5030B.

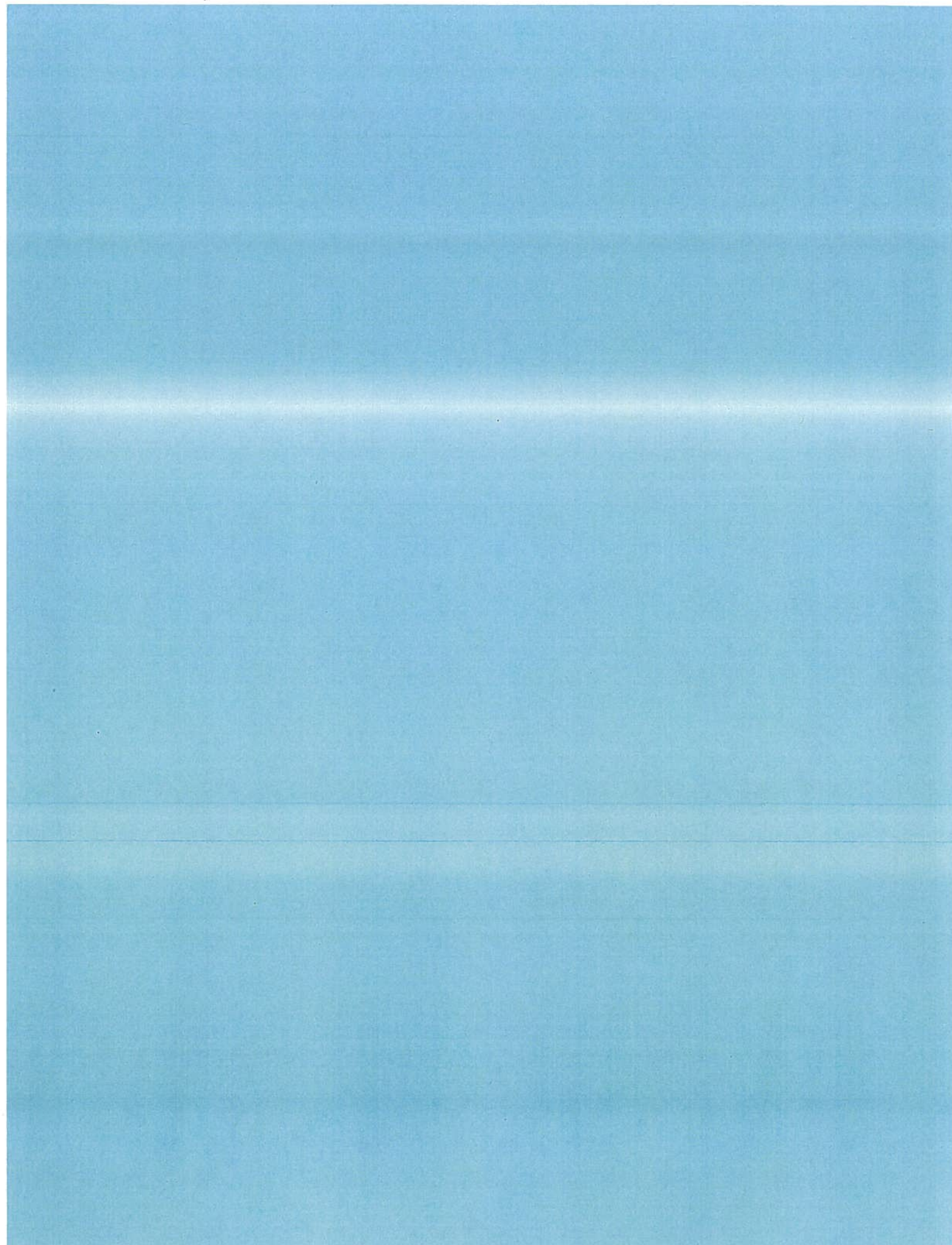
VOC = Volatile Organic Compounds

TCLP = Toxic Characteristics Leachate Procedure

SVOC = Semi-Volatile Organic Compounds

PNA = Polynuclear Aromatics

PCB = Polychlorinated Biphenyls



ATTACHMENT 1

FIELD ANALYTICAL STANDARD OPERATING PROCEDURES

DISSOLVED OXYGEN

Scope and Application: This method is applicable to surface water, wastewater and groundwater.

Method: Potentiometric

Reference: "Methods for Chemical Analysis of Water and Wastes:", EPA-600/4-79-020, revised March 1983, Method 360.1

Sensitivity: 0.1 mg/L as O₂

Optimum Range: 0.1 mg/L to 20 mg/L O₂

Sample Handling: Determined on site

Reagents and Apparatus:

1. Temperature compensated dissolved oxygen (DO) meter, Corning Check Mate System;
2. Zero oxygen standard;
3. DO sensor filling solution;
4. DO membrane replacement kit;
5. Distilled or deionized water in wash bottle.

Setting Up DO Sensor:

The sensor is shipped dry and must be filled before use.

1. Unscrew the membrane cap from sensor and fill using DO electrolyte.
2. Tap membrane cap gently to remove air bubbles. Gently screw cap onto probe body allowing surplus electrolyte to run out. (Caution: Do not overtighten)
3. Fit sensor to meter module.
4. Allow 30 minutes for polarization of electrode.

Calibration:

1. Remove wetting cap from tip of sensor. Switch on meter.
2. For first calibration point, place sensor in zero oxygen solution. Allow sufficient time for sensor to stabilize.
3. Move the sensor in a gentle circular motion.
4. Make sure sensor is immersed to a depth of 40 mm to cover the temperature sensing element.
5. Press "CAL" key. CAL 1 is displayed on meter and after endpointing, the display automatically updates to zero.
6. For second calibration point, hold sensor in air. Press "CAL" key. CAL 2 is displayed. After endpointing, the display automatically updates to 100% O₂.
7. To adjust oxygen calibration for salinity and barometric pressure, press "Mode" key. In mg/L O₂ mode, press "CAL" key and 100 is displayed. Use down arrow and up arrow on the keypad to adjust the display according to the salinity and barometric pressure tables contained on the operating instruction leaflet.

Procedure:

1. Calibrate meter using calibration procedure.
2. Pour the sample into clean sample jar or plastic cup.
3. Place sensor in sample. After following the immersion, stirring and stabilization steps referred to during calibration, press "READ" key to obtain sample result.
4. Record result in the field logbook.
5. Repeat steps 2 through 4 for each sample.

Quality Control:

1. Duplicate 1 out of 10 samples. If less than 10 samples are analyzed, a duplicate is still required. Duplicates must be within 15%.

If the results are outside of the control limits, rinse electrode 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 unable to recalibrate, replace sensor membrane.

2. Calibration check results must be within 10% of the true value. If the result is outside of 10%, 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 distilled or deionized water rinsed prior to analyses unless pre-cleaned sample jars are used.

Interferences:

Interferences in DO measurements generally occur due to membrane coating. Clean probe as specified in the sensor manual.

The presence of other gases such as chlorine, nitrous and nitric oxide, hydrogen sulfide and sulfur dioxide interfere with DO measurements. The sulfur based compounds will tarnish the electrodes resulting in sluggish or erratic measurements. Polishing the electrodes as specified in the operating manual will restore the performance of the meter. Recalibrate meter before analysis of next sample.

TURBIDITY

Scope and Application: This method is applicable to surface water, wastewater and groundwater.

Method: Nephelometric

Reference: "Methods for Chemical Analysis of Water and Wastes:", EPA-600/4-79-020, revised March 1983, Method 180.1

Sensitivity: 0.01 Nephelometric Turbidity Unit (NTU)

Optimum Range: 0 - 10; 0 - 100; 0-1,000 NTU

Sample Handling: Determined on site

Reagents and Apparatus:

1. Direct reading turbidity meter, HF Scientific Model DRT-15CE, HACH 2100P, and LaMotte;
2. Cuvettes with screw tops;
3. Battery charger;
4. 0.02 NTU (nominal) reference standard;
5. Distilled or deionized water in wash bottle.

Calibration Check and Operation

The turbidimeter has been calibrated by the manufacturer and electronic calibration using freshly prepared formazin standards should only be performed if the electronic printed circuit board, the photodetectors or the light source has been replaced.

The procedures for calibration checks and the operation of the meter follows:

1. For accurate measurements in the low range rotate the cuvettes in the well to obtain the minimum reading. Mark the cuvette with one of the adhesive dots provided with the instrument so that orientation of the cuvette will be identical each time it is placed in the instrument.
2. To operate the turbidimeter, switch to the "10" range and place the Reference Standard (0.02 NTU) in the optical well.

3. With the light shield in place over the well, adjust the Reference Adjust knob to cause the meter to read the reference standard value on the scale. The unit is now ready for use in either range.
4. To make a measurement of a sample, clean one of the cuvettes and fill to within approximately 3/4" from the top with sample. Place the top on the cuvette and carefully clean the outside surface of the cuvette with a lint free wiper such as KimWipes. Place the sample in the well and place the light shield over the well. Select the appropriate range for best readability. Record results in field logbook.
5. Repeat steps 3 and 4 for each sample.

Quality Control:

1. Duplicate 1 out of 10 samples. If less than 10 samples are analyzed, a duplicate is still required. Duplicates must be within $\pm 15\%$.

If the results are outside of the control limits, clean cuvettes 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).

2. Calibration check results must be $\pm 10\%$ of the true value. If the result is outside of $\pm 10\%$, clean cuvettes 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 distilled or deionized water rinsed prior to analyses.

Interferences:

Interferences in turbidity measurements are generally due to dirty or scratched cuvettes. Handle only the top one-third of the cuvettes and wipe clean using a lint-free wiper (KimWipes or equivalent).

OXIDATION-REDUCTION POTENTIAL (ORP)

Scope and Application: This method is applicable to surface water, wastewater and groundwater.

Method: Potentiometric

Reference: "Standard Methods for the Examination of Water and Wastewater", APHA, 18th edition, 1992, Method 2580B.

Sensitivity: 1 mV

Optimum Range: -1,500 to 1,500 mV

Sample Handling: Determined on site

Reagents and Apparatus:

1. ORP meter;
2. ORP electrode assembly;
3. Thermilinear thermistor temperature probe;
4. ZoBell Solution;
5. Distilled or deionized water in wash bottle.

Calibration:

1. Turn on the Water Quality Monitor and set the pH function switch to mV.
2. Connect the shorting cap attached to the to the mV input jack. The display should read 000 ± 2 mV. This indicates that the electronics are zeroed.
3. Detach the shorting cap and connect the to the mV input jack. If a pH electrode is not attached to the pH input jack, connect the shorting cap to it.
4. Attach the to the TEMP input jack.
5. Rinse with distilled or deionized water, followed by a rinse with a small amount of reconstituted ZoBell Solution.
6. Half fill a disposable 50 ml sample cup with ZoBell Solution and fully immerse the bulb and the end of the sheath. Allow the sensors to equilibrate, and note the reading.

7. The displayed mV values is not temperature compensated and should be corrected to 25°C at 1.3 mV/°C. The temperature coefficient is in reverse proportion to the temperature.
8. Correct the value to 25°C using the following equation:

$$\text{Actual Value mV} = \text{Display Value} + [(\text{Display Temp.} - 25^{\circ}\text{C}) \times (1.3 \text{ mV})]$$

Procedure:

1. Calibrate meter using calibration procedure.
2. Set up meter as outlined in the instruction manual.
3. Record temperature and ORP of the sample in the field logbook.
4. Correct ORP to 25°C using the formula presented above.
5. Record corrected ORP in the field logbook.
6. Repeat steps 3 through 6 for each sample.
7. Recheck calibration with ZoBell solution after every ten or fewer samples and after the last sample.
8. Store electrode in soaker bottle when not in use.

Quality Control:

1. Duplicate 1 out of 10 samples. If less than 10 samples are analyzed, a duplicate is still required. Duplicates must be ± 10 mV.

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 231 ± 10 mV. If the result is outside of this range, rinse electrodes and check solution again. If still outside the this range, recalibrate the meter and reanalyze all samples analyzed since the last in-control calibration.

Interferences:

Interferences in ORP measurements occur when the platinum electrode surface becomes coated. Clean the ORP electrode as follows:

1. Soft coatings should be removed by use of a wash bottle of water or by gently wiping with a soft cloth. Remove the bulb guard if necessary. Be careful not to scratch the platinum.
2. Hard coatings or organic chemicals should be removed by an appropriate chemical solvent, by gently scrubbing with a very fine cleansing powder such as "Softscrub", or by gently polishing with 600 grade wet silicon carbide paper. Wet a piece of the paper with water and gently polish the electrode with a circular twisting motion.

Note:

After cleaning the platinum surface, soak the electrode for a 8 to 24 hours in 4.0 pH buffer, then recheck it with ZoBell Solution before further use.

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 Wastes:", EPA-600/4-79-020, revised March 1983, Method 150.1

Sensitivity: 0.01 pH unit; 0.1 °C

Optimum Range: pH 1.00 to 12.00; temperature -5 to 50 °C

Sample Handling: Determined on site

Reagents and Apparatus:

1. Temperature compensated pH meter, Water Quality Monitoring System;
2. Combination pH electrode;
3. Thermilinear thermister temperature probe;
4. pH buffer solutions, pH 4.00, 7.00, and 10.00 (certified buffer solutions);
5. Distilled or deionized water in wash bottle.

Calibration:

1. Switch On/Off key to On. Before connecting the pH electrode, zero the electronics with the shorting cap attached to the meter. Turn on the meter and set the pH function switch to pH. Connect the shorting cap to the pH input jack and set the manual temperature compensation knob to 25°C. Adjust the CAL control to indicate 7.00 ±0.01 on the pH-mV display. Disconnect the shorting cap from the pH input and connect it to the mV input jack. The monitor is now zeroed.
2. Test the pH electrode for noise and offset as follows: Rinse the Temperature Probe with pH 7.00 buffer to remove any contaminants. Connect to the pH input jack and to the TEMP input jack. Pour pH 7.00 buffer into a 50 mL sample cup then immerse both of the sensors into the buffer at 25.0 ±0.1°C (use the °C display to confirm the temperature). Allow the sensors to equilibrate. A display value other than 7.00 shows electrode background noise and offset. The background noise and offset at pH 7.00 should not exceed ±0.2 pH units at 25°C. Replace pH probe if background noise exceeds this tolerance.

3. Set the function switch to pH ATC. Connect to the pH ATC input jack. The pH ATC function will not work unless it is connected to the pH ATC input.
4. Rinse the Temperature Probe with pH 7.00 buffer to remove any contaminants. Connect to the pH input jack and to the TEMP input jack. Pour pH 7.00 buffer into a 50 ml sample cup, immerse both of the sensor into the buffer. Allow the sensors the equilibrate in the buffer until a stable reading is obtained. Read the temperature and adjust the pH manual temperature compensation knob to the same value. Adjust the CAL control knob for 700 ± 0.01 pH units in the display and discard the buffer. Rinse the sensors with deionized or distilled water, followed by a rinse of the next desired buffer (typically pH 4.00 or 10.00). Half fill another disposable 50 ml sample cup with the next buffer for calibration and immerse the sensors. Allow the sensors to equilibrate until a stable reading is obtained. The temperature of the two buffers should not differ by more than $\pm 0.1^\circ\text{C}$. Adjust the SLOPE control until the display is within 0.01 pH units of the buffer's stated value. Discard the buffers. The pH system is now calibrated and ready for use.

Procedure:

1. Calibrate meter using calibration procedure.
2. Set up meter as outlined in the operating manual.
3. Pour the sample into clean sample jar or plastic cup.
4. Record temperature and pH of the sample in the logbook.
5. Rinse with water and pH 7.00 buffer.
6. Repeat steps 3 through 5 for each sample.
7. Recheck calibration with pH 7.00 buffer solution after every 10 or fewer samples and after the last sample.
8. Store pH electrode in soaker bottle when not in use.

Quality Control:

1. Duplicate 1 out of 10 samples. If less than 10 samples are analyzed, a duplicate is still required. Duplicates must be ± 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 ± 0.10 pH unit of the true value. If the result is outside of ± 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 water rinsed and distilled or deionized water rinsed prior to analyses.

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% HCl and deionized water rinse. 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: 0.1 mmhos/cm

Optimum Range: 0 - 100.0 mmhos/cm

Sample Handling: Determine on site

Reagents and Apparatus:

1. Conductivity meter - YSI Model Water Quality Monitoring System;
2. Conductivity Cell - YSI Model Flow-Through Conductivity Cell (K=5/cm);
3. Thermilinear Thermister - YSI Model Temperature Probe;
4. Deionized water;
5. Conductivity standard, 1.0mmho/cm @25°C.

Notes:

The conductivity meter is factory calibrated. The calibration is checked using a solution of known conductance.

Calibration Check

Connect the cell and the Temperature Probe, and remove them from the sample chamber. Set the conductivity function switch to 2 ATC. Rinse the inside and outside of the cell and the probe with about 1/3 the calibration solution. Place both of the sensors into the remainder of the solution in the bottle and allow them to come to temperature equilibrium. Make sure that the body is immersed so that the liquid level is half way up the knurled portion of the cell. Read the displayed value and determine if the cell/instrument is within specified accuracy. The displayed value is corrected to 25°C automatically and should be 1.000 ± 0.070 mmho/cm. If the value is not within specification replace cell.

Procedure:

1. Check calibration of meter.

2. Set up meter as outlined in the operating manual.
3. Before any conductivity cell is used, it should be soaked in distilled or deionized water for at least one hour. To make conductivity measurements, connect a YSI Flow-Through Conductivity Cell to the monitoring system. Set the conductivity function switch to 2 and observe the displayed value after the reading is stable. The display reads out in mmho/cm.
4. If the overrange signal (1._____) is displayed, the conductivity of the water being measured is greater than 1.999 mmho/cm. Reset the function switch to 20. If the overrange signal is still displayed, reset to 100. If the overrange signal is still displayed, either the conductivity is greater than 100.0 mmho/cm and the YSI Water Quality Monitor can not be used for conductivity determinations.
5. Record conductance readings in field logbook.
6. Repeat steps 3 through 5 for remaining samples.

Quality Control:

1. The quality control calibration check standard must be analyzed initially, after every 10 or fewer samples and after the last sample. If less than 10 samples are analyzed, the calibration standard is still required to be analyzed. The standard 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 $\pm 15\%$ 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 probe.

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

Sample Handling: Determined on site

Reagents and Apparatus:

1. HNu Photoionizer or Photovac Microtip Photoionization Detector;
2. Calibration gas Isobutylene 100ppm;
3. Calibration apparatus and tubing;
4. Battery charger.

Calibration:

A) Calibration of the HNu

1. Connect the analyzer to the regulator and calibration gas 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.

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. The samples for VOC headspace screening will be prepared in the field by filling a 4-ounce soil jar to one-half its volume and sealing with a teflon-lined closure. The remaining sample will be placed in the appropriate jars for the analyses required.
3. Allow the sample for VOC headspace screening to remain at ambient temperature for a minimum of 10 minutes. This will allow for VOCs in the soil to reach equilibrium in the headspace of the jar.
4. Remove the lid of the soil jar slightly and insert the probe of the meter into the headspace.
5. Take the highest reading from the meter or readout of the instrument.
6. Record the reading in the field logbook.
7. Recheck calibration with calibration gas after a minimum of every 10 samples and after the last sample.

Quality Control:

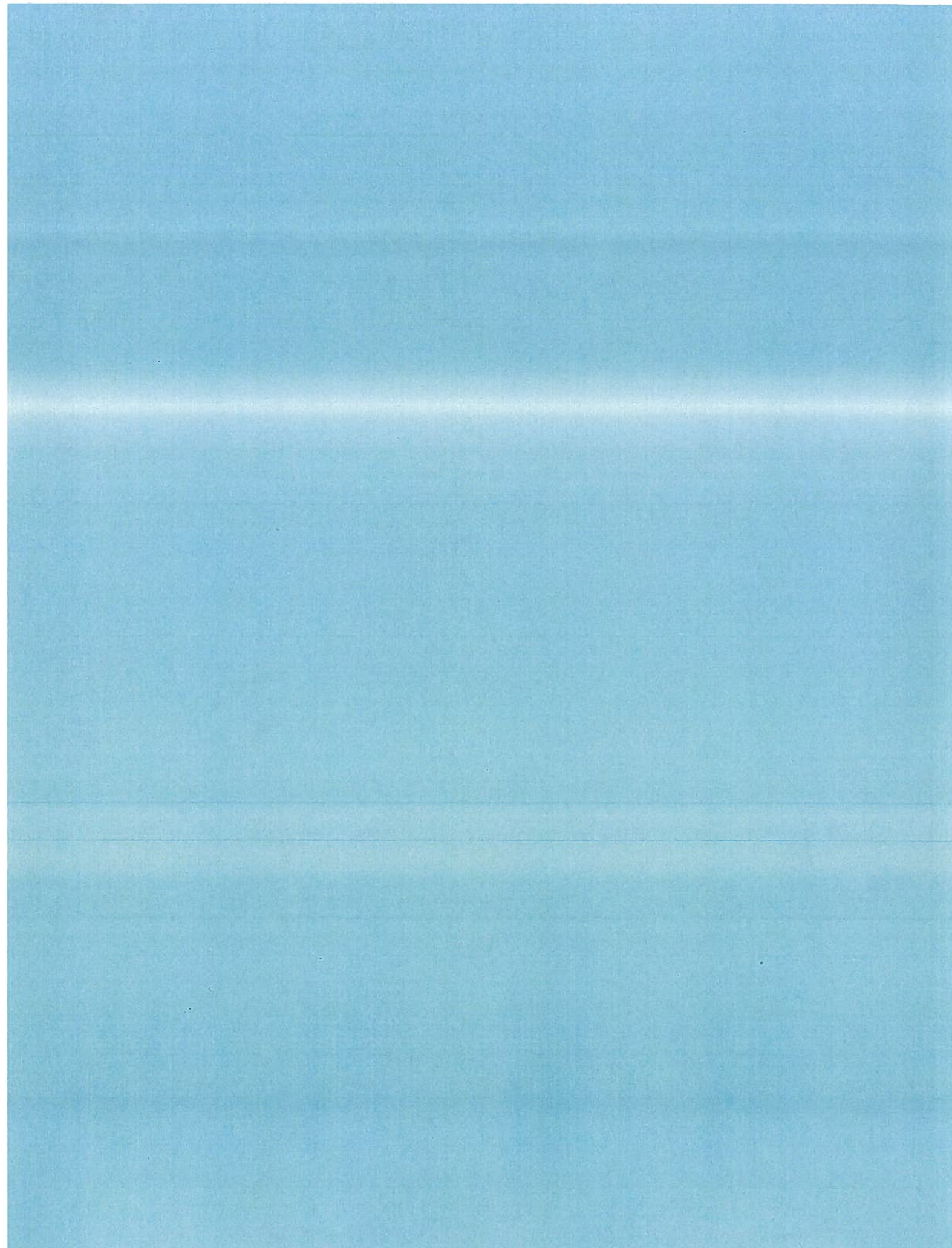
1. Calibration check results must be ± 10 percent of the true value. If the result is outside of ± 10 percent, recalibrate the meter as specified above.

SOP Number:	FP-VOC-02
Revision Number:	0
Date:	11-15-02
Page Number:	4 of 3

2. Duplicate samples are not analyzed since the headspace VOC readings will vary considerably as the 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.



ATTACHMENT 2

LABORATORY ANALYTICAL STANDARD OPERATING PROCEDURES

Controlled Copy No. _____
Implementation Date 9-26-07

SOP No. CORP-QA-0010
Revision No. 3
Revision Date: 09/19/07
Page 1 of 12

TESTAMERICA STANDARD OPERATING PROCEDURE

TITLE: NONCONFORMANCE AND CORRECTIVE ACTION SYSTEM

(SUPERSEDES: REVISION 2, DATED 06/15/99)

Approved by: Norothy J. Keson 9/21/07
Quality Assurance Manager Date

Approved by: Carl Byler 9/21/07
Laboratory Director Date

Approved by: Mark Bruce 9/24/07
Technical Director Date

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TABLE OF CONTENTS

<u>1.</u>	<u>Purpose</u>	3
<u>2.</u>	<u>Responsibilities</u>	3
<u>3.</u>	<u>SAFETY</u>	5
<u>4.</u>	<u>Procedure</u>	5
<u>5.</u>	<u>Definitions</u>	10
<u>6.</u>	<u>Miscellaneous</u>	11

1. PURPOSE

- 1.1. The purpose of this document is to establish procedures for the identification and documentation of nonconformances and the corrective actions taken as a result of these events. The TestAmericaQuality requires documentation of instances of deviations from established control limits, approved standard operating procedure (SOPs), or client-specified requirements. The Nonconformance Memo (NCM) described in this procedure is used to document deviations from TestAmerica policies and procedures and documented client specifications including root causes and corrective actions taken to remedy the nonconformance. The NCM may be stored in electronic form in a database, or maintained using defined printed forms.
- 1.2. This document applies to procedures, services, analytical data, reports, or materials purchased by the laboratory or supplied by the laboratory to its clients. Nonconformances related to sample receiving activities may be documented separately from the NCM process by use of the Cooler Receipt Form (CRF) as described in a facility-specific SOP. Regardless of the type of system is used to handle the sample receiving and client-related issues, including holding time violations (HTVs), the system must include and emphasize the immediate notification of the Project Manager (PM). This will allow the PM to initiate immediate client notification and resolution of how to proceed. See Section 5, Definitions, for further clarification of application.
- 1.3. Nonconformances can be identified by TestAmerica laboratory employees in the course of their daily operations or by external parties (i.e., customers and representatives of customers) through reviews of records, audit, or proficiency testing.

2. RESPONSIBILITIES

- 2.1. **Laboratory Associate:** During the course of their work, all employees are responsible for identifying and documenting problems, using a Nonconformance Memo, that might affect the quality of TestAmerica's product. They should also identify or attempt to seek out possible measures to correct the problem. By signing or initialing laboratory notebooks, forms, bench sheets, data reports, and other quality-related documents, associates are verifying that procedures have been followed. Any deviation that might render a measurement suspect shall be documented.
- 2.2. **Group Leader/Team Leader/Supervisor** (for purposes of this SOP, the term "Supervisor" will be used): Each Supervisor is responsible for the review of NCMs to ensure that problems which might affect quality are adequately described and that personnel are assigned to correct them. Supervisors review hardcopy or electronic versions of NCMs and forward them to the appropriate Project Manager. Together with Project Managers

and Quality Assurance personnel, Supervisors are responsible for determining the appropriateness of planned corrective actions.

- 2.3. **Project Manager (PM):** The Project Manager is responsible for relaying project requirements to staff so that special project requirements are understood and nonconformances recognized. The Project Manager communicates conformance problems to clients and documents decisions made with clients. The Project Manager ensures that short-term corrective actions for routine analytical QC failures are completed. An example would be making sure that reparation and analysis of a sample was done. The Project Manager can and must withhold final reports to clients until corrective actions agreed to with the client have been completed.
- 2.4. **QA Manager:** The Quality Assurance manager or his or her designee is responsible for reviewing all initiated NCMs to ensure that actions taken are appropriate, and assisting in resolving QA/QC discrepancies. The QA staff will maintain a nonconformance tracking system to guarantee that each nonconformance is brought to closure. The system will also be used to monitor for trends that might indicate long-term quality problems. Systematic problems are investigated, NCMs issued and reviewed, and spot audits conducted to ensure that long-term corrective actions have been successfully completed. If review of an area reveals a significant problem with data quality, the Quality Assurance manager has the authority and responsibility to stop production in that laboratory area.
- 2.5. **Operations/Systems Manager :** The operations/systems manager shall ensure that corrective actions are correct and have been implemented. The operations/systems manager shall document this review and concurrence by signing the NCM as the responsible manager, if QA-required, for a specific corrective action. Along with the laboratory manager, the operations/systems manager shall emphasize the importance of quality requirements and require all employees to report any problem that might adversely affect the quality of work.
- 2.6. **Laboratory Manager:** The laboratory manager shall emphasize the importance of quality requirements and require all employees to report any problem that might adversely affect the quality of work. The laboratory manager is also responsible for the implementation of the NCM system in the laboratory.
- 2.7. **Corporate QA Director:** The TestAmerica Quality Assurance Director should be notified of any continuing nonconformances that are not properly addressed by operations or where the root cause cannot be identified.

3. SAFETY

- 3.1. Normal office dependent safety precautions must be taken in performing this SOP. If personnel are required to perform any portion of the procedure in laboratory areas, appropriate personal protective equipment and precautions must be utilized.
- 3.2. Procedures shall be carried out in a manner that protects the health and safety of all TestAmerica associates.
- 3.3. 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.

4. PROCEDURE

- 4.1. When to Initiate a Nonconformance Memo
 - 4.1.1. Lab associates are to initiate an electronic nonconformance memo (NCM) whenever procedures, services, data, reports, electronic disk deliverables (EDDs), or standard materials deviate from established specifications. All nonconformances with the exception of matrix-related failures require an NCM (see definitions of nonconformance, anomalies, and deficiencies in Section 5 and Section 1.2 for exceptions).
 - 4.1.2. All standard operating procedures (SOPs) shall be followed. By signing or initialing laboratory notebooks, bench sheets, data reports, and other quality-related documents, employees are verifying that the SOPs have been followed with the exceptions of the pre-approved deviations (as described in QAPPs Quality Assurance Summaries, or equivalent systems). Any intentional deviation from an SOP must be pre-approved by the QA manager and Supervisor. Any deviation from a SOP or client requirement not previously approved must be documented using the NCM process.
 - 4.1.3. An NCM is to be completed for each instance of a nonconformance. A single NCM can be used for a single event affecting multiple lot numbers and samples, but normally a separate NCM would be initiated for different nonconformance issues. If the nonconformance involves projects for multiple Project Managers, then the NCM will need to be routed to each Project Manager.
 - 4.1.4. Laboratories QuantIMS shall use the NCM database application developed for this purpose, **Clouseau**.

4.2. How to Process the NCM using Clouseau

4.2.1. Initiating the NCM

- 4.2.1.1. While properly logged in to a PC where Clouseau has been installed, start the Clouseau program.
- 4.2.1.2. At the Main Menu, select Create NCM.
- 4.2.1.3. Enter the information required at the top of the form. At a minimum select the Production Area, Classification (anomaly or deficiency), NCM Type, and NCM Description.
- 4.2.1.4. For NCMs that affected samples, indicate the relevant batch numbers, lots, samples, and tests by using the Tests/Samples option within Clouseau.
- 4.2.1.5. Complete the Comments/Details and Corrective Action sections of the Create NCM Form. These sections may be filled out jointly with the Supervisor. Consult the Project Manager or the operations/systems manager and QA Manager if the Supervisor and associate are uncertain of corrective actions. Be objective and specific. Include enough information that decisions to approve the NCM can be made easily (include pertinent QC information). Include enough information that details can be inserted directly into the Case Narrative section of the client report.
 - 4.2.1.5.1. Design corrective actions to correct the immediate problem (short-term corrective actions) and to minimize the possibility of its recurrence (long-term corrective actions). Examples of corrective actions are modifications to nonconforming procedures, repair or replacement of deficient equipment, training personnel, and reanalysis of any affected samples.
 - 4.2.1.5.2. Where operational corrective actions are required, they shall be supported with reference to recovery data, control charts, or other documentation.
- 4.2.1.6. If the corrective action involved retraining, the training must be documented with the signatures of the trainees and submitted to the QA staff before the NCM is considered closed.

4.2.1.7. Save the NCM using the appropriate command in Clouseau. When presented with the Send E-Mail form, the Supervisor of the affected Production Area, the Project Manager, and the QA Department will be listed in the "Notify Now" box. Verify that these names are correct. If any personnel should be informed that are not listed, add their names to the "Notify Now" box by double-clicking on their names in the Personnel box.

4.2.1.8. Initiate the NCM notification process by selecting SEND EMAIL.

4.2.2. Supervisory Review and Approval

4.2.2.1. The Supervisor will receive notification of the NCM via e-mail. The Supervisor must log in to a computer workstation where Clouseau has been installed, and run Clouseau.

4.2.2.2. Using the Review NCM form, select the NCM to be reviewed and click on OPEN.

4.2.2.3. With the NCM on screen, review the information provided. Add necessary comments or corrective actions to the appropriate areas. If the corrective action has not been determined, the situation must be referred to the Project Manager and the operations/systems manager for resolution to ensure client requirements can be satisfied. The QA staff should be consulted if there are questions as to how to proceed. If the above input was not needed, the operations/systems manager does not need to have every NCM routed to him or her. If, upon receipt and review of the NCM by the QA staff, it is felt the operations/systems manager needs to be made aware of the issues, the QA staff will notify the operations/systems manager using the Under Review/Send Email options of Clouseau.

4.2.2.4. If the nonconformance is hardware/equipment related, the item shall be nonconformance tagged and segregated, if possible, to ensure that it is not used until repaired. Refer to Section 4.3.5.

4.2.2.5. The Supervisor will be responsible for the completion of the corrective action unless otherwise indicated. Enter the name of the person responsible for performing the corrective action if other than the Supervisor. This is Operations' commitment to rectify the problem. This will be verified by the QA staff and/or the Operations Managers.

4.2.2.6. The Supervisor selects the "Approved" option on the Review NCM form and selects SAVE. This initiates the NCM routing process. If the NCM is for a holding time violation, a Project Manager ***must*** be notified ***immediately***.

4.2.3. Project Manager Review, Client Notification, and Project Documentation

4.2.3.1. The Project Manager shall determine if client notification is required to either assist in the definition of corrective action or to notify the client of problems related to sample analysis. The Project Manager shall indicate using the Client Notification Form in Clouseau whether client notification is required or not.

4.2.3.2. Record the result of the client contact or leave blank if client contact was not required. Indicate the date the contact was made. This must be done whether client notification is completed or not.

4.2.3.3. With the NCM on screen, review the information provided. Add necessary comments or corrective actions to the appropriate areas. Notify the Supervisor of any changes made to the corrective action plan. The Project Manager's review of the NCM serves as documentation that the PM has read and reviewed the NCM and is aware of the corrective action plan.

4.2.3.4. The Project Manager selects the "Approved" option on the Review NCM form and selects SAVE. This initiates the NCM routing process.

4.2.3.5. If it is found that erroneous analytical data (e.g., from data validation comments or phone requests, etc.; inaccurate chromatograms, spectra, calculations, or final reports) have been released by the laboratory, this fact must be documented on an NCM and forwarded to the QA office. Prior to making the corrections, proper documentation shall be filled out and turned in to the QA staff if corrections are needed in the database (QuantIMS). The original data shall be marked as unusable and maintained for historical purposes. The corrective action shall include prompt client notification and issuance of amended reports.

4.2.3.6. After the QA staff has closed the NCM, the NCM is stored in the Clouseau program for future reference

4.2.4. Quality Assurance Review and Trending

- 4.2.4.1. The QA staff shall review all NCMs for conformance with standard laboratory practices.
- 4.2.4.2. NCMs will be reviewed to ensure that the corrective action was completed.
- 4.2.4.3. The Clouseau reporting and tracking system will be used to monitor for repetitive failures that might indicate systematic problems. Tracking records would (when applicable) include:
 - NCM log number
 - Date initiated
 - Project number
 - Lab sample ID numbers
 - Method or parameter
 - Nonconformance description
 - Corrective action required
 - Characterization as an anomaly or deficiency
 - Closure of NCM
- 4.2.4.4. The QA staff shall identify repetitive quality issues that may be systematic in nature and may require corrective actions to prevent recurrence. Recurrent technical or Information Technology problems shall be referred to the appropriate technical group for corrective actions. Correction of systematic problems could take the form of modifications of nonconforming procedures, repair or replacement of deficient equipment, training or replacement of personnel. Findings and corrective actions from these investigations or audits shall also be documented. Resolution of corrective actions for systematic problems must be documented by the responsible laboratory area, along with supporting evidence.
- 4.2.4.5. The QA staff shall conduct follow-up assessments to confirm that correction of systematic problems is successful.
- 4.2.4.6. The approval of the QA manager or designee is required in Clouseau to indicate that the NCM has been closed.
- 4.2.4.7. Clouseau will be the official database of all NCMs whether closed or pending for review and closure.
- 4.2.5. Instrument/Equipment Nonconformance Tag

- 4.2.5.1. Instruments and equipment which habitually fail to meet calibration criteria or are out of service due to needed repair or other reasons must be marked with a clearly visible tag or sign indicating the nonconforming condition. (See Example in Attachment B.)
- 4.2.5.2. If the reason for the nonconformance tag caused sample data to be impacted, initiate an NCM (electronic or hard copy). Identify the instrument by name and/or identification number and briefly describe the problem.
- 4.2.5.3. Upon saving the NCM, the NCM number indicated by Clouseau shall be recorded in the instrument's maintenance log.
- 4.2.5.4. The corrective action will be to either permanently remove the instrument from service or to have the instrument repaired. If an instrument is repaired, its reliability must be demonstrated through successful recalibration before the nonconformance can be closed. The nonconformance tag remains in effect during the demonstration period. Record the back to control information in the instrument maintenance logbook. Reference the successful calibration on the tag and return the tag to the QA staff for closure of the NCM

5. DEFINITIONS

- 5.1. **Nonconformance:** an unplanned deviation from an established protocol or plan. The deviation may be the result of TestAmerica actions--then termed a **deficiency**, or the result of events beyond the control of TestAmerica--then termed an **anomaly**.

A nonconformance exists when:

- 5.1.1. Any laboratory QC sample (e.g., method blank, laboratory control sample, duplicate laboratory control sample, matrix spike, matrix spike duplicate, and surrogate spike) component result is outside established control limits and demonstrate a **systematic** deficiency. Any matrix spike or matrix spike duplicate or sample related QC outside of established control limits attributed to matrix effects must be documented, but an NCM is not required.
- 5.1.2. A procedure is not performed as described in the applicable SOP or QA Policy, **except** in cases where the procedure has been performed according to a client-specified document TestAmerica has agreed to follow (e.g., EPA SOWs and QAPPs).

- 5.1.3. A practice or procedure is not performed as described according to a client or project document that TestAmerica has agreed to follow.
- 5.1.4. Purchased materials or services are determined to be defective and their use would effect data quality.
- 5.1.5. Holding time violations occur regardless of what or whose actions caused them.
- 5.1.6. A formal NCM is not required for routine instrument maintenance and malfunctions, which can be documented in instrument maintenance logbooks.
- 5.2. ***Corrective action:*** Measures taken to rectify conditions adverse to quality and, where possible, to prevent their reoccurrence.
 - 5.2.1. Corrective actions may vary from reporting the data as is—with appropriate documentation—to a complete reevaluation and restructure of a system.
 - 5.2.2. Many corrective actions can be implemented immediately; however, some will take time to implement.

6. MISCELLANEOUS

- 6.1. Associated Reference Documents
 - 6.1.1. TestAmerica Laboratory Quality Manual (LQM), current revision.
 - 6.1.2. Clouseau Program Documentation.
- 6.2. Appendices
 - 6.2.1. Attachment A: Instrument/Equipment Nonconformance Tag Form

ATTACHMENT A

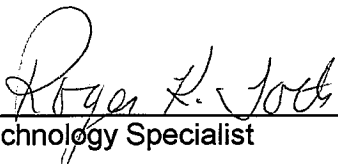
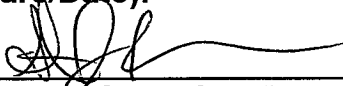
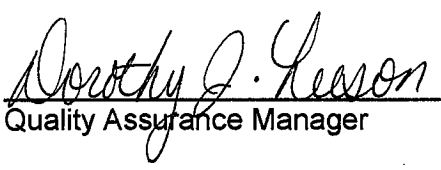
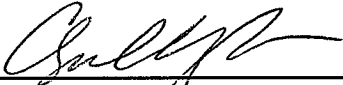
EXAMPLE

INSTRUMENT/EQUIPMENT NONCONFORMANCE TAG FORM

TESTAMERICA	
CAUTION	
DO NOT USE	
NONCONFORMING ITEM	
NCM NUMBER _____	
AFFECTED ITEM _____	

_____	_____
ANALYST	DATE
WORK MAY NOT PROCEED ON THIS ITEM UNTIL SUCCESSFUL CALIBRATION IS DOCUMENTED.	

Title: Acid Digestion for Aqueous Samples
[Method: SW846 and MCAWW 200 Series Methods]

Approvals (Signature/Date):	
 Technology Specialist	 Health & Safety Coordinator
12-14-07 Date	12-14-07 Date
 Quality Assurance Manager	 Laboratory Director
12/13/07 Date	12/14/07 Date
 Technical Director	
12/14/07 Date	

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Revision No. 1.6

Revision Date: 02/07/07

Page 1 of 29

STL STANDARD OPERATING PROCEDURE

TITLE: ACID DIGESTION OF AQUEOUS SAMPLES BY SW846 AND MCAWW 200 SERIES METHODS

(Supersedes: Revision 1.5, Dated 12/07/04)

Reviewed by:	<u>Roger K. Toeh</u>	<u>3-6-07</u>
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	Technical Director	Date

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TABLE OF CONTENTS

1. SCOPE AND APPLICATION	3
2. SUMMARY OF METHOD	4
3. DEFINITIONS	4
4. INTERFERENCES	5
5. SAFETY	5
6. EQUIPMENT AND SUPPLIES	7
7. REAGENTS AND STANDARDS	8
8. SAMPLE COLLECTION, PRESERVATION AND STORAGE	9
9. QUALITY CONTROL	10
10. CALIBRATION AND STANDARDIZATION	12
11. PROCEDURE	13
12. DATA ANALYSIS AND CALCULATIONS	16
13. METHOD PERFORMANCE	16
14. POLLUTION PREVENTION	17
15. WASTE MANAGEMENT	17
16. REFERENCES	17
17. MISCELLANEOUS (TABLES, APPENDICES, ETC. . .)	18

LIST OF APPENDICES:

APPENDIX A - TABLES	21
APPENDIX B - CONTAMINATION CONTROL GUIDELINES	28

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 (ICP/MS) using the MCAWW 200 series methods (NPDES) and SW846 Methods 3005A, 3010A, and 3020A.
- 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 is used to prepare surface and groundwater samples for total recoverable and dissolved metals determination by ICP.
- 1.5. MCAWW Method 200.7 is used to prepare surface water, domestic and industrial waste samples for 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.
- 1.7. MCAWW Method 200.7 is used to prepare surface water and wastes that contain suspended solids for total metals analysis by ICP.
- 1.8. All matrices require digestion prior to analysis with the exception of analyses for dissolved metals in filtered and acidified aqueous samples. Although digestion is not specifically required by the method, some clients and regulators do require digestion of dissolved samples and this must be clarified before project initiation.
- 1.9. SW846 Method 3020A is used to prepare surface and groundwater samples for total recoverable and dissolved metals determination by ICP/MS.

- 1.10. MCAWW Method 200.8 is used to prepare surface and groundwater samples for total recoverable and dissolved metals determination by ICP/MS.

2. **SUMMARY OF METHOD**

- 2.1. Method 3005A / Method 200.7 - Preparation for Total Recoverable or Dissolved Metals Analysis by ICP Spectroscopy

2.1.1. A representative aliquot of sample is heated with nitric and hydrochloric acids and substantially reduced in 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 Spectroscopy

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 hydrochloric acid, filtered (if necessary) and brought up to volume.

- 2.3. Method 3020A/Method 200.8 – Preparation for total recoverable or dissolved metals analysis by ICP/MS.

2.3.1. A representative aliquot of sample is heated with nitric acid and until the digestate has been reduced to a low volume. The sample is cooled, filtered (if necessary), and brought up to volume.

3. **DEFINITIONS**

Additional definitions of terms used in this SOP may be found in the glossary of the LQM.

- 3.1. Dissolved Metals: Those elements which pass through a 0.45 um membrane. (Sample is acidified after filtration).
- 3.2. Suspended Metals: Those elements which are retained by a 0.45 um membrane.
- 3.3. Total Metals: The concentration determined on an unfiltered sample following digestion.
- 3.4. Total Recoverable Metals: The concentration determined on an unfiltered sample following treatment with hot, dilute mineral acid.

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.
- 4.2. The entire work area, including the bench top and fume hood, should 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 and silica from the glassware will migrate into the sample solution during and following sample processing. For critical low level determinations of boron and silica, only quartz and/or plastic labware should 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. If physical interferences are present, they should be documented.
- 4.5. Visual interferences or anomalies (such as foaming, emulsions, precipitates, etc.) 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.

5. **SAFETY**

- 5.1. Employees must abide by the policies and procedures in the Corporate Safety 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 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
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 will be removed and discarded; other gloves will 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 should 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**. Therefore, unless they are known to be non-hazardous, all samples should be opened, transferred and prepared in a fume hood, or under other means of mechanical ventilation. 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 will 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 STL 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.

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.
- 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. Centrifugation equipment (if desired method of removing particulates is centrifugation).
- 6.8. Graduated cylinder or equivalent capable of measuring 50 mL within 3% accuracy.
- 6.9. Analytical balance capable of accurately weighing to the nearest 0.01 grams.
- 6.10. Repipetors or suitable reagent dispensers.
- 6.11. Calibrated automatic pipettes with corresponding pipette tips or Class A glass volumetric pipettes.
- 6.12. Class A volumetric flasks.
- 6.13. pH indicator strips (pH range 0 - 6).
- 6.14. Plastic digestate storage bottles.

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.
- 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% HNO₃. This acid (5 mL of concentrated HNO₃ 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 ICP/MS 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 (LCSW) 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 ICP/MS LCS and matrix spike preparations.
 - 7.8. Nitric acid (HNO_3), concentrated, trace metal grade or better.
 - 7.9. 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.
 - 7.10. Hydrochloric acid (HCl), concentrated, trace metal grade or better.
 - 7.11. 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 may be stored in either plastic or glass. If boron or silica are 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 should 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.

Note: If a sample being analyzed for dissolved metals is found to contain sediment the analyst should contact their supervisor or group leader. The client should be notified of the problem to decide how to treat the sample.

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

Prior to analysis of any analyte using any method contained within this SOP 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 MDL is determined using seven replicates of reagent water, spiked with all the analytes of interest, that have been carried through the entire analytical procedure. MDL's must be redetermined on an annual basis in accordance with 40 CFR Part 136 Appendix B requirements as detailed in STL QA Policy S-Q-003 and SOP NC-QA-0021. The spike level must be between the calculated MDL and 10X the MDL to be valid. The result of the MDL determination must be below the STL reporting limit.

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 should 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 (CORP-MT-0001).

- 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, a 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.
- 9.3. 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. MS/MSD are not included in the sample count unless there are multiple sets of MS/MSD per batch. In other words, the first MS/MSD are not counted; all additional MS and MSDs are counted as samples.
- 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 SOP's. 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. Aqueous method blanks are prepared by taking 50 mL of reagent water through the appropriate procedure as described in Section 11.
- 9.4.2. TCLP method blanks are prepared by taking 50 mL of leachate fluid through the appropriate procedure as described in Section 11.
- 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. On going 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 SOP's. Corrective action when LCS results fail to meet control limits will be repreparation and reanalysis of the batch. Refer to Section 7.3 and 7.4 for instructions on preparation of the aqueous LCS spike solution.

9.5.1. 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 ICP/MS 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.

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 (DQO's) may require the use of sample duplicates in place of or in addition to MS/MSD's. 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.

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 ICP/MS 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.

9.6.3. 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 MS/MSD criteria.

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, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using a Nonconformance Memo. If contractually required, the client shall be notified. The Nonconformance Memo shall 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.
- 11.3. All digestion procedures must be 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.
- 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. 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.
- 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.
- 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 and transfer 50 mL of the sample into a beaker.

11.9.3. Measure two extra aliquots of sample selected for the MS/MSD analysis. Spike each aliquot with the appropriate spiking solutions (Sections 7.3-7.5 and 9.6).

11.9.4. Measure and transfer 50 mL of reagent water into a beaker for the method blank.

11.9.5. Measure and transfer 50 mL of reagent water into a beaker for the LCS and add the appropriate spiking solutions (Sections 7.3-7.5 and 9.6).

11.10. Method 3005A / Method 200.7 - Preparation for Total Recoverable or Dissolved Metals Analysis by ICP

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.

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.

Note: If any samples in a preparation batch are filtered, the method blank and LCS associated with that batch must also be filtered.

Note: In place of filtering, the samples, after dilution and mixing, may be centrifuged or allowed to settle by gravity overnight to remove insoluble material.

11.10.6. Rinse container and filter paper with reagent water to ensure complete sample transfer.

11.10.7. Adjust the final volume/mass to 50 mL or 50 g with reagent water. 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 3.0mL of concentrated HNO₃.
- 11.11.2. Cover with ribbed watch glass.
- 11.11.3. Place container on hotblock 90-95°C, and evaporate for 4-5 hours or to low volume of 15-20 mL, while ensuring that no portion of the bottom of the beaker is allowed to go dry.

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.

Note: If any samples in the QC batch are filtered the method blank and LCS associated with that batch must also be filtered.

Note: In place of filtering, the samples, after dilution and mixing, may be centrifuged or allowed to settle by gravity overnight to remove insoluble material.

- 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. The sample is now ready for analysis.

11.12. Method 3020A / Method 200.8 - Preparation for Total or Total Recoverable Metals by ICP/MS

- 11.12.1. To the sample container, add 1.5 mL of concentrated HNO₃.
- 11.12.2. Cover with ribbed watch glass.

- 11.12.3. Place beaker on hotplate 90-95°C and evaporate to low volume of 15-20 mL while ensuring that no portion of the bottom of the beaker is allowed to go dry.

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.

- 11.12.4. Filter sample, if insoluble materials are present, through Whatman 41 filter paper.

Note: If any samples in the QC batch are filtered the method blank and LCS associated with that batch must also be filtered.

Note: In place of filtering, the samples, after dilution and mixing, may be centrifuged or allowed to settle by gravity overnight to remove insoluble material.

- 11.12.5. Rinse beaker and filter paper with reagent water to ensure complete sample transfer.

- 11.12.6. Adjust final volume to 50 mL with reagent water. The sample is now ready for analysis.

- 11.12.7. ready for analysis.

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. In general, the matrix spike recovery should fall within +/- 20 % and the matrix spike duplicates should compare within 20% RPD. Method blanks must meet the criteria specified in determinative SOPs. The laboratory control samples should recover within 20% of the true value until in house control limits are established. 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 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. This method allows for the proportional reduction of sample and reagent volumes to decrease waste generation.

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 Safety Manual 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 of STL. 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 3020A.
- 16.1.2. Methods for the Chemical Analysis of Water and Waste (MCAWW), 1983.
- 16.1.3. CORP-MT-0001, Inductively Coupled Plasma-Atomic Emission Spectroscopy, Spectrometric Method for Trace Element Analysis of Water and Wastes, Method 6010A, 6010B, and Method 200.7.
- 16.1.4. Corporate Quality Management Plan (QMP), current version.
- 16.1.5. STL Laboratory Quality Manual (LQM), current version.
- 16.1.6. STL Corporate Safety Manual, M-E-0001 and STL North Canton Facility Addendum and Contingency Plan, current version.
- 16.2. Associated SOPs and Policies, latest version
 - 16.2.1. QA-003, STL QC Program.
 - 16.2.2. QA-004, Rounding and Significant Figures.
 - 16.2.3. Glassware Washing, NC-QA-0014
 - 16.2.4. Statistical Evaluation of Data and Development of Control Charts, NC-QA-0018
 - 16.2.5. Method Detection Limits and Instrument Detection Limits, S-Q-003 and NC-QA-0021
 - 16.2.6. Supplemental Practices for DoD Project Work, NC-QA-0016
 - 16.2.7. Standards and Reagents, NC-QA-0017
- 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. Chapter 1 of SW-846 states that the method blank should 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.

17.1.1.2. 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 the 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 11.12.3 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.

17.1.2.2. The scope of 3010A has been expanded to include silver based on comparison studies with 7760A. Method 3010A consistently demonstrated improved accuracy and precision over Method 7760A in the matrices tested (reagent water, surface water and TCLP leachate) up to a concentration of 1 ppm silver.

17.1.3. Modifications Specific to Method 3020A

17.1.3.1. Section 11.13.3 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.

17.1.4. Modifications Specific to MCAWW Methods

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).

17.2. Documentation and Record Management

17.2.1. The preparation benchsheet should, at a minimum, include the following information:

- Preparation date, analyst name, matrix, prep type (ICP or ICP/MS), Method reference.
- Sample ID with initial weight/volume and final weight/volume.
- Standards documentation (source, lot, volume added).
- Analyst
- Reagents

APPENDIX A

TABLES

TABLE I. Approved Preparation Method Analytes - SW846

ELEMENT	Symbol	CAS Number	3005A	3010A	3020A
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	X
Cadmium	Cd	7440-43-9	X	X	X
Calcium	Ca	7440-70-2	X	X	
Chromium	Cr	7440-47-3	X	X	X
Cobalt	Co	7440-48-4	X	X	X
Copper	Cu	7440-50-8	X	X	
Iron	Fe	7439-89-6	X	X	
Lead	Pb	7439-92-1	X	X	X
Magnesium	Mg	7439-95-4	X	X	
Manganese	Mn	7439-96-5	X	X	
Molybdenum	Mo	7439-98-7	X	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	X
Vanadium	V	7440-62-2	X	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.

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.

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.

TABLE IV. ICP/MS Aqueous LCS and Matrix Spike Levels

ELEMENT	Working LCS/MS Standard (mg/L)	Aqueous LCS/MS Level* (ug/L)
Aluminum	100	1000
Antimony	10	100
Arsenic	10	100
Barium	10	100
Beryllium	10	100
Cadmium	10	100
Calcium	100	1000
Chromium	10	100
Cobalt	10	100
Copper	10	100
Iron	100	1000
Lead	10	100
Magnesium	100	1000
Manganese	10	100
Molybdenum	10	100
Nickel	10	100
Potassium	100	1000
Selenium	10	100
Silver	10	100
Sodium	100	1000
Strontium	10	100
Thallium	10	100
Vanadium	10	100
Zinc	10	100
Boron	10	100
Tin	10	100
Titanium	10	100
Zirconium	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 (7.4) to 50 mL of sample.

TABLE V. TCLP Reporting Limits, Regulatory Limits and Matrix Spike Levels

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 (7.4) to 50 mL of sample.

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 - CORP-MT-0001	Redigest and reanalyze 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 - CORP-MT-0001	Redigest and reanalyze 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 - CORP-MT-0001	Reprep not required unless preparation error suspected.
Matrix Spike Duplicate	See Matrix Spike	Refer to determinative SOPs: - NC-MT-0002 - CORP-MT-0001	See Corrective Action for Matrix Spike.

APPENDIX B

CONTAMINATION CONTROL GUIDELINES

APPENDIX B. CONTAMINATION CONTROL GUIDELINES

The following procedures are strongly recommended to prevent contamination:

All work areas used to prepare standards and spikes should be cleaned before and after each use.

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 must be kept scrupulously clean.

Powdered or Latex Gloves must not be used in the metals laboratory since the powder contains silica and zinc, as well as other metallic analytes. Only vinyl or nitrile gloves should be used in the metals laboratory.

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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Implementation Date: 7-18-07

SOP No. CORP-MT-0005NC

Revision No. 2.6

Revision Date: 07/10/07

Page 1 of 37

TESTAMERICA NORTH CANTON STANDARD OPERATING PROCEDURE

**TITLE: PREPARATION AND ANALYSIS OF MERCURY IN AQUEOUS SAMPLES BY
COLD VAPOR ATOMIC ABSORPTION, SW846 7470A AND MCAWW 245.1**

(SUPERSEDES: REVISION 2.5 DATED 01/08/04)

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PREPARATION AND ANALYSIS OF MERCURY IN AQUEOUS
SAMPLES BY COLD VAPOR ATOMIC ABSORPTION, SW-846
METHOD 7470A AND MCAWW METHOD 245.1

SOP No. CORP-MT-0005NC

Revision No. 2.6

Revision Date: 07/10/07

Page 2 of 37

TABLE OF CONTENTS

1. SCOPE AND APPLICATION	3
2. SUMMARY OF METHOD	3
3. DEFINITIONS	4
4. INTERFERENCES.....	4
5. SAFETY	5
6. EQUIPMENT AND SUPPLIES	7
7. REAGENTS AND STANDARDS	8
8. SAMPLE COLLECTION, PRESERVATION AND STORAGE	9
9. QUALITY CONTROL.....	9
10. CALIBRATION AND STANDARDIZATION	13
11. PROCEDURE.....	14
12. DATA ANALYSIS AND CALCULATIONS.....	17
13. METHOD PERFORMANCE	18
14. POLLUTION PREVENTION	19
15. WASTE MANAGEMENT	19
16. REFERENCES	19
17. MISCELLANEOUS (TABLES, APPENDICES, ETC. . .).....	20

LIST OF APPENDICES:

APPENDIX A - TABLES	26
APPENDIX B - MSA GUIDANCE	30
APPENDIX C - TROUBLESHOOTING GUIDE.....	33
APPENDIX D- CONTAMINATION CONTROL GUIDELINES.....	35
APPENDIX E- PREVENTATIVE MAINTENANCE.....	37
APPENDIX F – INSTRUMENT SET-UP	39

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 SW-846 Method 7470A and MCAWW Method 245.1.
- 1.2. The associated LIMs method codes are BL (Method 245.1) and O8 (Method 7470A).
- 1.3. CVAA analysis provides for the determination of total mercury (organic and inorganic). The combination of the oxidants, potassium permanganate and potassium persulfate, 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 (see CORP-MT-0007NC) 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. 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.
- 1.7. 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 and nitric acids. Organic mercury compounds are oxidized with potassium permanganate and potassium persulfate 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. Dissolved Metals: Those elements which pass through a 0.45 um membrane. (Sample is acidified after filtration).
- 3.2. Suspended Metals: Those elements which are retained by a 0.45 um membrane.
- 3.3. Total Metals: The concentration determined on an unfiltered sample following digestion.
- 3.4. Refer to the glossary in the Laboratory Quality Manual (LQM), latest 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. Specific selection of reagents may be required to avoid introduction of contaminants.
- 4.2. Potassium permanganate, which is used to breakdown 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.
- 4.3. 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.
- 4.4. Chlorides can cause a positive interference. Sea waters, brines and industrial effluents high in chlorides require additional permanganate (as much as 25 mL) because, during the oxidation step, chlorides are converted to free chlorine, which also absorbs radiation at 253.7 nm. Care must be taken to ensure that free chlorine is absent before the mercury is reduced and swept into the cell. This is accomplished by adding excess hydroxylamine reagent (25 mL) and purging the sample headspace before stannous chloride is added. Both inorganic and organic mercury spikes have been quantitatively recovered from seawater using this technique.

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.

- 4.5. Interference from certain volatile organic materials that absorb at this wavelength may also occur. 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.
- 4.6. Samples containing high concentrations of oxidizing 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.
- 4.7. 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.

5. SAFETY

- 5.1. Employees must abide by the policies and procedures in the Corporate Safety 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 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 Poison	1 Mg/M3-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	2 ppm-TWA	Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison. Inhalation of vapors can cause

	Oxidizer Poison	4 ppm-STEL	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.
Potassium Permanganate	Oxidizer	5 Mg/M3 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.
Mercury (1,000 ppm in Reagent)	Oxidizer Corrosive Poison	0.1 Mg/M3 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.
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.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

5.3. 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 will be removed and discarded; other gloves will be cleaned immediately.

5.4. Exposure to chemicals must be maintained **as low as reasonably achievable**. Therefore, unless they are known to be non-hazardous, all samples should be opened,

transferred and prepared in a fume hood, or under other means of mechanical ventilation. Solvent and waste containers will be kept closed unless transfers are being made. All samples with stickers that read "Caution/Use Hood!" **must** be opened in the hood.

- 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 a Laboratory Supervisor and the EH&S Coordinator.
- 5.6. Do not look directly into the beam of the Hg lamp. The UV light that these lamps radiate is harmful to the eyes.
- 5.7. Cylinders of compressed gas must be handled with caution, in accordance with local regulations. It is recommended that, wherever possible, cylinders are located outside the laboratory and the gas led to the instrument through approved lines.
- 5.8. 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 a 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 Aeration Tubing: A straight glass frit having a coarse porosity and Tygon tubing is used for the transfer of mercury vapor from the sample bottle to the

absorption cell and return. The tubing must be inert and mercury-free.

6.2.7 Drying device to prevent condensation in cell. The lamp is positioned to shine on the absorption cell maintaining the air temperature in the cell about 10 °C above room temperature. Other drying devices that achieve the same purpose are also acceptable (i.e., Gortex filter).

6.3. 8oz. HDPE Plastic bottles.

6.4. Nitrogen or argon gas supply, welding grade, or equivalent.

6.5. Calibrated automatic pipettes or Class A glass volumetric pipettes.

6.6. Class A volumetric flasks.

6.7. Thermometer (capable of accurate readings at 95 °C).

6.8. 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 (in 10% HNO₃) 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.

7.3. Working mercury standard (0.1 ppm): Take 1 mL of the stock mercury standard (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% HNO₃. This acid (150 uL of concentrated HNO₃) must be added to the flask/bottle before the addition of the stock standard aliquot.

7.4. The calibration standards listed in Table I must be prepared fresh daily from the working standard (7.3) by transferring 0, 0.2, 0.5, 1.0, 5.0 and 10.0 mL aliquots of the working mercury standard into 100 mL flasks and diluting to volume with reagent water.

- 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 (HNO₃), concentrated, traces metal grade or better.
- 7.8. Sulfuric acid (H₂SO₄), concentrated, traces metal grade or better.
- 7.9. Stannous chloride solution: Add 50 g of stannous chloride and 25 mL of concentrated hydrochloric acid to a 500mL volumetric flask and bring to volume with deionized water.
- 7.10. Sodium chloride-hydroxylamine hydrochloride solution: Add 240 g of sodium chloride and 240 g of hydroxylamine hydrochloride to every 2000 mL of reagent water.
- 7.11. Potassium permanganate, 5% solution (w/v): Dissolve 100 g of potassium permanganate for every 2000 mL of reagent water.
- 7.12. 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 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. Refrigeration is not required. Preservation must be verified prior to analysis.

9. **QUALITY CONTROL**

- 9.1. Table II (Appendix A) provides a summary of quality control requirements including type, frequency, acceptance criteria and corrective action.
- 9.2. Initial Demonstration of Capability

- 9.2.1 Prior to the analysis of any analyte using 7470A or the 245.1, the following requirements must be met.
- 9.2.1.1 Method Detection Limit (MDL) - An MDL must be determined for each analyte/matrix prior to the analysis of any samples. The MDL is determined using seven replicates of reagent water, spiked with all the analytes of interest, that have been carried through the entire analytical procedure. MDLs must be predetermined on an annual basis in accordance with 40 CFR Part 136 Appendix B requirements. The spike level must be between the calculated MDL and 10X the MDL to be valid. The result of the MDL determination must be below the TestAmerica North Canton reporting limit.
- 9.2.2 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.
- 9.2.2.1 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 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. 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. 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 should not contain any analyte of interest at or above the reporting limit 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).

- Re-preparation 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. **Such action must be addressed in the project narrative.**
- If the above criteria are not met and reanalysis 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 aqueous LCS must be processed with each preparation batch. The LCS is used to monitor the accuracy of the analytical process. On going 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.

- 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.
- In the event that an MS/MSD analysis is not possible, 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.
- Corrective action will be re-preparation and reanalysis of the batch unless the client agrees that other corrective action is acceptable.

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 (DQO's) may require the use of sample duplicates in place of or in addition to MS/MSD's. 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 A).

- 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 75 - 125 % recovery for Method 7470A, 70 – 130% for Method 245.1, and 20% RPD 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 reanalysis 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 4x the spike level for that analyte, the recovery data is 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.”
- If an MS/MSD is not possible due to limited sample volume, then a laboratory control sample duplicate (LCSD) should be analyzed. The RPD of the LCS and LCSD must be compared to the matrix spike RPD limits.

- 9.7. Initial Calibration Verification (ICV/ICB) - Calibration accuracy is verified by analyzing a second source standard (ICV). For Method 7470A, 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. If either the ICV or ICB fail to meet criteria, the analysis should be terminated, the problem corrected and the instrument re-calibrated. (See Section 11.2.8 for required run sequence). If the cause of the ICV or ICB failure was not directly instrument related the corrective action will include re-preparation of the ICV, ICB, CRA, CCV, and CCB with the calibration curve.
- 9.8. 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 must be 80 – 120%. A CCB is analyzed immediately following each CCV. (See Section 11.2.8 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 ICV/CCV and ICB/CCB pairs.

- 9.9. 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. Recovery must be $\pm 50\%$ 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. (See Section 11.2.8 for the required run sequence.)
- 9.10. Method of Standard Addition (MSA) -This technique involves adding known amounts of standard to one or more aliquots of the sample prior to preparation. This technique compensates for a sample interferent that may enhance or depress the analyte signal, thus producing a different slope from that of the calibration standards. It will not correct for additive interferences, which cause a baseline shift. Refer to Section 11.2.9 for additional information on when full 4 point MSA is required as well as Appendix B for specific MSA requirements.

10. CALIBRATION AND STANDARDIZATION

- 10.1. Calibration standards must be processed through the preparation procedure as described in Section 11.1.
- 10.2. 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.
- 10.3. Set up the instrument with the operating parameters recommended by the manufacturer and listed in Appendix F. Allow the instrument to become thermally stable before beginning calibration (approximately 30 minutes of warm-up is required).
- 10.4. Calibrate the instrument according to instrument manufacturer's instructions, using a minimum of five standards and a blank. One standard must be at the TestAmerica North Canton 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.
- 10.5. The calibration curve must have a correlation coefficient of ≥ 0.995 or the instrument shall be stopped and recalibrated prior to running samples. Sample results can not be reported from a curve with an unacceptable correlation coefficient. NOTE: If any digested standard does not meet criteria, all Ohio VAP samples associated with that curve will be re-digested.
- 10.6. Refer to Section 9.0 for calibration verification procedures, acceptance criteria and corrective actions.

11. PROCEDURE

11.1. Sample Preparation:

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. Transfer 0, 0.2, 0.5, 1.0, 5.0 and 10.0 mL aliquots of the working standard (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.

11.1.2 Transfer 100 mL of well-mixed sample or standard to a clean sample digestion bottle.

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.

11.1.3 Add 5 mL of concentrated H_2SO_4 and 2.5 mL of concentrated HNO_3 mixing after each addition.

11.1.4 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 reanalysis may be required.

Note: When performing analyses using automated vs. manual techniques the sample dilution resultant from the addition of more than the original aliquot of permanganate solution must be compensated for by the addition of the same volume of permanganate to all other associated samples and standards in the run. In instances, where this is not feasible, the addition of excess reagent can be addressed through mathematical correction of the results to account for the resultant dilution effect.

11.1.5 Add 8 mL of potassium persulfate solution and heat for two hours in a water bath at 90 - 95 °C.

11.1.6 Cool samples.

11.2. Sample Analysis:

- 11.2.1 Refer to the Appendix F of this SOP for detailed setup and operation protocols.
- 11.2.2 When ready to begin analysis, add 6 mL of sodium chloride-hydroxylamine hydrochloride solution to the samples to reduce the excess permanganate (the permanganate has been reduced when no purple color remains).
- 11.2.3 Automated determination: Follow instructions provided by instrument manufacturer.
- 11.2.4 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. Calibration using computer or calculation based regression curve fitting techniques on concentration/response data is acceptable.
- 11.2.5 All measurements must fall within the defined calibration range to be valid. Dilute and reanalyze all samples for analytes that exceed the highest calibration standard.
- 11.2.6 If the sample results are negative and the absolute value of the negative result is greater than the reporting limit, the sample must be diluted and reanalyzed.
- 11.2.7 The samples must be allowed to cool to room temperature prior to analysis or a decrease in the response signal can occur.
- 11.2.8 The following run sequence is consistent with 7470A and 245.1.
 - Instrument Calibration
 - ICV
 - ICB
 - CRA
 - CCV
 - CCB
 - 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 to apply to Methods 7470A and 245.1.

11.2.9 For TCLP samples, full four point MSA will be required if all of the following conditions are met:

- 1) recovery of the analyte in the matrix spike is not at least 50%,
- 2) the concentration of the analyte does not exceed the regulatory level, and,
- 3) the concentration of the analyte is within 20% of the regulatory level.

The reporting and matrix spike levels for TCLP analyses are detailed in Table I (Appendix A). Appendix B provides guidance on performing MSA analyses. For TCLP mercury determinations, MSA spikes must be added prior to sample preparation.

11.3. To facilitate the early identification of QC failures and samples requiring rerun it is strongly recommended that sample data are reviewed periodically throughout the run.

11.4. Guidelines are provided in the appendices on procedures to minimize contamination of samples and standards, preventive maintenance and troubleshooting.

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 an 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.

11.6. One time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using a Nonconformance Memo and is approved by a Technical Specialist and QA Manager. If contractually required, the client shall be notified. The Nonconformance Memo shall be filed in the project file.

11.7. Any unauthorized deviations from this procedure must also be documented as a nonconformance, with a cause and corrective action described.

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 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

12.6. The LCS percent recovery is calculated according to the following equation:

$$\%R = 100 \left(\frac{Found(LCS)}{True(LCS)} \right)$$

12.7. Appropriate factors must be applied to sample values if dilutions are performed.

12.8. Sample results should be reported with up to three significant figures in accordance with the TestAmerica North Canton significant figure policy.

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. Method performance is determined by the analysis of method blanks, laboratory control samples, matrix spike and matrix spike duplicate samples. The matrix spike recovery should fall within +/- 25 % and the matrix spike duplicates should compare within 20% RPD. The method blanks must meet the criteria in Section 9.3. The laboratory control sample should recover within 20% of the true value until in house limits are established.

13.3. Training Qualification:

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. This method allows for the proportional reduction of sample and reagent volumes to decrease waste generation.

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 Safety Manual 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 an annual refresher training.

15.3. Waste Streams Produced by the Method

15.3.1 **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 Corporate Quality Management Plan (QMP), current version.

16.1.4 TestAmerica Laboratory Quality Manual (LQM), current version.

16.2. Associated SOPs and Policies, latest version

16.2.1 QA Policy, QA-003.

16.2.2 Glassware Washing, NC-QA-0014.

16.2.3 Statistical Evaluation of Data and Development of Control Charts, NC-QA-0018.

16.2.4 Method Detection Limits and Instrument Detection Limits, NC-QA-0021.

16.2.5 Supplemental Practices for DoD Project Work, SOP, NC-QA-0016.

16.2.6 Preparation and Analysis of Mercury in Aqueous Samples by Cold Vapor
Atomic Absorption, Method 245.1 and CORP-MT-0006NC, current version.

16.2.7 Standards and Reagents, Sop NC-QA-0017.

17. **MISCELLANEOUS (TABLES, APPENDICES, ETC. . .)**

17.1. Modifications/Interpretations from reference method.

17.1.1 Modifications from both 7470A and 245.1.

17.1.1.1 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.

17.1.1.2 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."

17.1.2 Modifications from Method 7470A

- 17.1.2.1 Chapter 1 of SW-846 states that the method blank should 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.

17.1.3 Modifications from 245.1

- 17.1.3.1 Method 245.1 states that standards are not heated. TestAmerica North Canton prepares heated standards for this method.

APPENDIX A

TABLES

**TABLE I . MERCURY REPORTING LIMITS, CALIBRATION STANDARD*, QC
STANDARD AND SPIKING LEVELS (MG/L)**

Standard Aqueous RL	0.0002
TCLP RL	0.002
Std 0	0
Std 1/CRA	0.0002
Std 2	0.0005
Std 3	0.001
Std 4	0.005
Std 5	0.010
ICV	0.0025
LCS/CCV	0.005
Aqueous MS	0.001
TCLP MS	0.005

TABLE II. Summary Of Quality Control Requirements

QC PARAMETER	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
ICV	Beginning of every analytical run.	90-110 % recovery.	Terminate analysis; Correct the problem; Recalibrate or reprep with calibration curve. (See Section 9.6).
ICB	Beginning of every analytical run, immediately following the ICV.	The result must be within +/- RL from zero if the samples are at or above the reporting limit	Terminate analysis; Correct the problem; Recalibrate or reprep with calibration curve. (See Section 9.6).
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	50-150% recovery	Rerun to verify; or correct problem and recalibrate or reprep with the calibration curve (See Sec. 9.8)
CCV	Every 10 samples and at the end of the run.	Recovery for Method 245.1 = 95-105% following a calibration. All other CCV for 245.1 are 80-120%. Recovery for Method 7470A = 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. (See Section 9.7).

CCB	Immediately following each CCV.	The result must be within +/- RL from zero. If the CCB 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 CCB or reprep with calibration curve. (See Section 9.7).
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 re-digestion (See Section 9.3).	Re-digest and reanalyze samples. Note exceptions under criteria section. See Section 9.3 for additional requirements.

TABLE II. Summary of Quality Control Requirements (Continued)

QC PARAMETER	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
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. For Method 245.1, the LCS must be 85-115%.	Terminate analysis; Correct the problem; Re-digest and reanalyze all samples associated with the LCS (see Section 9.4).
Matrix Spike	One per sample preparation batch of up to 20 samples.	75 - 125 % recovery or in-house control limits. 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 > 4x the spike added. (see Section 9.5) For TCLP see Section 11.2.9
Matrix Spike Duplicate	See Matrix Spike	75 - 125 % recovery or in-house control limits; RPD $\leq$ 20%. (See MS)	See Corrective Action for Matrix Spike.

APPENDIX B
MSA GUIDANCE

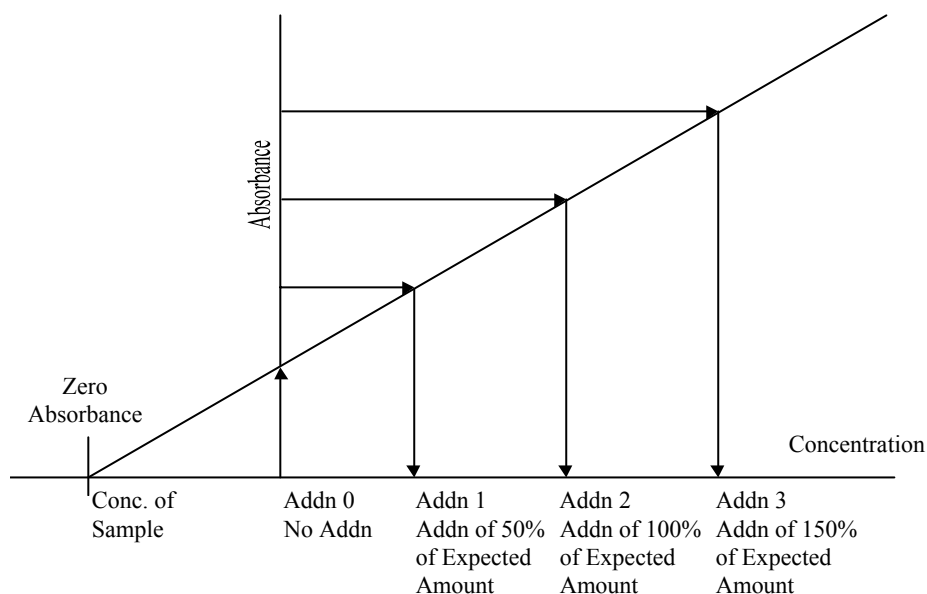
APPENDIX B. MSA GUIDANCE

Method of Standard Addition

Four equal volume aliquots of sample are measured and known amounts of standards are added to three aliquots. The fourth aliquot is the unknown and no standard is added to it. The concentration of standard added to the first aliquot should be 50% of the expected concentration. The concentration of standard added to the second aliquot should be 100% of the expected concentration and the concentration of standard added to the third aliquot should be 150% of the expected concentration. The volume of the unspiked and spiked aliquots should be the same (i.e., the volume of the spike added should be negligible in relation to the volume of sample).

To determine the concentration of analyte in the sample, the absorbance (or response) of each solution is determined and a linear regression performed. On the vertical axis the absorbance (or response) is plotted versus the concentrations of the standards on the horizontal axis using 0 as the concentration of the unspiked aliquot. An example plot is shown in Figure 1. When the resulting line is extrapolated back to zero absorbance, the point of interception of the horizontal axis is the concentration of the unknown. Calculate the correlation coefficient (r) and the x-intercept (where $y=0$) of the curve. The concentration in the digestate is equal to the negative x-intercept.

Figure 1



- For the method of standard additions to be correctly applied, the following limitations must be taken into consideration.
- The plot of the sample and standards must be linear over the concentration range of concern. For best results, the slope of the curve should be similar to that of a plot of the aqueous standard curve.
- The effect of the interference should not vary as the ratio of the standard added to the sample matrix changes.

APPENDIX C
TROUBLESHOOTING GUIDE

APPENDIX C. TROUBLESHOOTING GUIDE

Problem	Possible Cause
Poor or No Absorbance or Sensitivity Check failed	Incorrect wavelength Dirty windows Window loose Etched or dirty optics Wrong lamp Bad lamp Not enough or no sample introduced Empty sample cup Incorrectly made standards Gas leak Tubing blockage
Erratic Readings	Source lamp not aligned properly Lamp not prewarmed Injection tip partially clogged Contaminated reagents Contaminated glassware Drying tube saturated Bad lamp Injection tip hitting outside of tube Injection tip coated or not set properly Leak in sample tubing Power fluctuations Air bubbles in tubing
Lamp Won't Light	Lamp cable not plugged in Lamp power set at 0 Lamp is dead Power supply fuse is blown Short in cord
Standards reading twice or half normal absorbance or concentration	Incorrect standard used Incorrect dilution performed Dirty cell
Background Correction Light Blinking	Background screen or attenuator faulty

APPENDIX D
CONTAMINATION CONTROL GUIDELINES

APPENDIX D. CONTAMINATION CONTROL GUIDELINES

The following procedures are strongly recommended to prevent contamination:

All work areas used to prepare standards and spikes should be cleaned before and after each use.

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 must be kept scrupulously clean.

Powdered gloves must not be used in the metals laboratory since the powder contains silica and zinc, as well as other metallic analytes. Alternatively, vinyl or nitrile gloves should be used in the metals laboratory.

Glassware should 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.

APPENDIX E

PREVENTIVE MAINTENANCE

APPENDIX E. PREVENTIVE 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.

Cold Vapor Atomic Absorption (Leeman PS 200II)

Daily	As Needed
Check nitrogen flow.	Check Hg lamp intensity.
Check tubing.	Clean lens.
Check drain.	Check aperture.
	Replace drying tube.
	Change Hg lamp.
	Check liquid/gas separator.

APPENDIX F
INSTRUMENT SET UP

Hg Analysis (Leeman PS200II)

SYSTEM INITIALIZATION AND WARM UP

1. F1 Menu
2. Instrument
 - a. TASKMASTER
 - b. #4 Wake System Up Enter

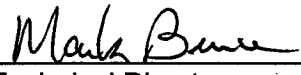
The warming up period takes approximately 10 minutes.

TO SET UP INSTRUMENT FOR ANALYSIS

1. F1 Menu
2. Autosampler
 - A. Rack Entry
 - B. Edit (ex. Rack 1), Enter
 - C. Cup ID - Enter (clears sample #'s)
 - D. Extended ID- type in matrix of sample (water or solid), Enter.
 - E. Press Insert Key and move cursor with arrows to cup ID and begin typing labels.
 - F. F3 Print Screen
3. Press F2 Macro key and type in analyst's first name - Enter
 - A. Enter folder name - ex. HG10306, 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 3 times to begin analysis.

**Title: GAS CHROMATOGRAPHIC ANALYSIS BASED ON METHODS
8000B, 8021B, 8081A, 8082, 8151A, 8015B, AND 615**

Approvals (Signature/Date):			
	1/18/08		1-18-08
Technology Specialist	Date	Health & Safety Coordinator	Date
	1/18/08		1/17/08
Quality Assurance Manager	Date	Laboratory Director	Date
	1/18/08		
Technical Director	Date		

This SOP was previously identified as SOP CORP-GC-0001NC, Rev 5.8, dated 02/06/06

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TABLE OF CONTENTS

1.	Scope and Application.....	4
2.	Summary of Method.....	4
3.	Definitions.....	4
4.	Interferences.....	4
5.	Safety.....	4
6.	Equipment and Supplies.....	6
7.	Reagents and Standards.....	6
8.	Sample Preservation and Storage.....	6
9.	Quality Control.....	7
10.	Calibration and Standardization.....	9
11.	Procedure.....	15
12.	Data Analysis and Calculations.....	17
13.	Method Performance.....	20
14.	Pollution Prevention.....	21
15.	Waste Management.....	21
16.	References.....	21
17.	Miscellaneous.....	21

List of Appendices:

Appendix A	Analysis of Volatile Organics based on Method 8021B
Appendix B	Analysis of Organochlorine Pesticides based on Method 8081A
Appendix C	Analysis of Organochlorine Pesticides and PCBs based on Method 8082
Appendix D	Analysis of Phenoxy Acid Herbicides based on Method 8151A
Appendix E	Total Petroleum Hydrocarbons Based on 8015B

LIST OF TABLES

Table A1	Standard Analyte Lists for 8021B
Table A2	Recommended conditions for Method 8021B
Table A3	Surrogate and Internal Standard Concentrations for Aqueous and Low Level Soil Samples, Method 8021B
Table A4	Concentrations for LCS and MS/MSD Compounds, Low Level Soil and Aqueous, Method 8021B
Table B1	Standard Analyte and Reporting Limits, Method 8081A
Table B2	Recommended Conditions, Method 8081A
Table B3	Calibration levels, Method 8081A
Table B4	Column Degradation Evaluation Mix, Method 8081A
Table B5	LCS/Matrix Spike and Surrogate Levels, Method 8081A
Table B6	LCS/Matrix Spike and Surrogate Levels for TCLP, Method 8081A
Table B7	Suggested Analytical Sequence, Method 8081A
Table C1	Standard Analyte List, Method 8082
Table C2	Instrumental Conditions, Method 8082
Table C3	Calibration Standards, Method 8082
Table C4	LCS/Matrix Spike and Surrogate Levels, Method 8082
Table C5	Michigan Analyte List and Reporting Limits, Method 8082
Table C6	Suggested Analytical Sequence, Method 8082
Table D1	Standard Analyte List, Method 8151A
Table D2	Instrumental Conditions, Method 8151A
Table D3	LCS/Matrix Spike and Surrogate Levels, Method 8151B
Table E1	Suggested GC Temperature Program for TPH Analysis
Table E2	Reporting Limits for TPH Analysis

1. SCOPE AND APPLICATION

This SOP describes procedures for analysis of organic analytes by Gas Chromatography (GC). The procedures are based on SW-846 methodology and are applicable for measurements made to comply with the Resource Conservation and Recovery Act (RCRA). Individual analytes and methods are described in the appendices. Appendix E describes procedures for the analysis of petroleum hydrocarbons by SW-846 8015B methodology

2. SUMMARY OF METHOD

In general, semivolatile analytes in aqueous samples are prepared for analysis using continuous or separatory funnel liquid / liquid extraction or solid phase extraction (SOP # CORP-OP-0001NC). Solid samples are prepared using sonication, Soxhlet or automated Soxhlet (PCB only) (SOP # CORP-OP-0001NC). Volatile analytes are prepared for analysis using purge and trap methodology (Appendix A).

After the initial preparation step, the sample is introduced to the GC and concentrations of target analytes are measured by the detector response within a defined retention time window, relative to the response to standard concentrations. Internal or external standardization procedures are used as specified in the method appendices.

3. DEFINITIONS

Definitions of terms used in this SOP may be found in the glossary of the TestAmerica North Canton Laboratory Quality Manual (LQM), current version.

4. INTERFERENCES

Contamination by carryover can occur when a low concentration sample is analyzed after a high concentration sample. Co-elution of target analytes with non-targets can occur, resulting in false positives or biased high results. In particular, this is a problem with non-selective detectors such as the Flame Ionization Detector (FID). See the appendices for interferences specific to individual tests and suggested corrective actions.

5. SAFETY

- 5.1. Employees must abide by the policies and procedures in the Corporate Safety Manual and this document.
- 5.2. Eye protection that prevents splash, laboratory coat, and appropriate gloves must be worn while samples, standards, solvents, and reagents are being handled. Refer to the TestAmerica North Canton Corporate Safety Manual for a complete description of personal protection equipment. Cut-resistant gloves must be worn doing any other task that presents a strong possibility of getting cut. Disposable gloves that have become contaminated will be removed and discarded; other gloves will be cleaned immediately. Latex, Nitrile and vinyl gloves all provide adequate protection against the methanol used in this method.
- 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 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 (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.
Acetone	Flammable	1000 ppm-TWA	Inhalation of vapors irritates the respiratory tract. May cause coughing, dizziness, dullness, and headache.
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.
Methylene Chloride	Carcinogen Irritant	25 ppm-TWA 125 ppm-STEEL	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.
Sulfuric Acid	Corrosive Oxidizer Dehydrator Poison Carcinogen	1 Mg/M3-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.
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.			

- 5.4. Exposure to chemicals must be maintained **as low as reasonably achievable**; therefore, unless they are known to be non-hazardous, all samples must be opened, transferred, and prepared in a fume hood, or under other means of mechanical ventilation, where possible. 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 will be kept closed unless transfers are being made.
- 5.5. Opened containers of neat standards will be handled in a fume hood.
- 5.6. Sample extracts and standards, which are in a flammable solvent, shall be stored in an explosion-proof refrigerator.
- 5.7. When using hydrogen gas as a carrier, all precautions listed in the CSM shall be observed.
- 5.8. Standard preparation and dilution shall be performed inside an operating fume hood.
- 5.9. 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.

- 5.10. 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.
- 5.11. 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.

6. EQUIPMENT AND SUPPLIES

- 6.1. An analytical system complete with a gas chromatograph is required. A data system capable of measuring peak area and/or height is required. Recommended equipment and supplies for individual methods are listed in each method appendix.

7. REAGENTS AND STANDARDS

7.1. Stock Standards

- 7.1.1. Stock standards are purchased as certified solutions or prepared from pure solutions. Stock standards for method 8021B are stored at -10 to -20°C. Other stock standard solutions are stored as recommended by the manufacturer. All stock standards must be protected from light. Stock standard solutions should be brought to room temperature before using.
- 7.1.2. Semivolatile stock standard solutions must be replaced after one year. Stock standards of gases must be replaced at least every week, unless the acceptability of the standard is demonstrated (Less than 20% drift from the initial calibration is an acceptable demonstration). Other volatile stock standards must be replaced every six months or sooner if comparison with check standards prepared from an independent source indicates a problem.
- 7.1.3. Expiration times for all standards are measured from the time the standard is prepared or from the time that the standard ampoule is opened, if the standard is supplied in a sealed ampoule. If vendor-supplied standard has an earlier expiration date then that date is used. Refer to SOP NC-QA-0017, Standards and Reagents, for additional information.

7.2. Calibration Standards

7.2.1. Volatile Calibration Standards

- 7.2.1.1. The procedure for preparation of volatile standards is given in Appendix A.

7.2.2. Semivolatile Calibration Standards

- 7.2.2.1. Semivolatile calibration standards are prepared as dilutions of the stock standards. Surrogates and internal standards are used as specified in the method appendices. Semivolatile calibration solutions must be refrigerated at $\leq 6^{\circ}\text{C}$ and protected from light. The standards must be replaced at least every six months or sooner if comparison with check standards indicates a problem.

7.3. Gases for carrier and make-up: Hydrogen, Helium, Nitrogen, Zero Air.

7.4. Quality control (QC) Standards

- 7.4.1. QC standards (matrix spiking and LCS standards) are prepared and stored in the same way as calibration standards. They must be made from a stock independent from the calibration standards.

8. SAMPLE PRESERVATION AND STORAGE

- 8.1 Semivolatile extracts must be refrigerated at $\leq 6^{\circ}\text{C}$ and analyzed within 40 days of the end of the extraction. Volatile sample storage conditions and holding times are given in Appendix A.

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, 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 an extracted standard at the reporting limit and a single point calibration.

9.2. Batch Definition

Batches are defined at the sample preparation stage. Batches should be kept together through the whole analytical process as far as 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.

9.2.1. Quality Control 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. Laboratory generated QC samples (Blank, LCS, MS/MSD) do not count towards the maximum 20 samples in a batch. Field QC samples are included in the batch count. In some cases, at client request, the MS/MSD may be replaced with a matrix spike and sample duplicate. If insufficient sample is available for an MS/MSD a LCSD may be substituted.

9.3. Control Limits

In-house historical control limits may be determined for surrogates, matrix spikes, and laboratory control samples (LCS). These limits must be determined at least annually. The recovery limits are mean recovery ± 3 standard deviations, unless that limit is tighter than the calibration criteria, in which case limits may be widened. Project or program specific control limits may be used in place of in-house limits. Refer to policy QA-003 for more details.

- 9.3.1. These limits do not apply to dilutions (except for tests without a separate extraction), but surrogate and matrix spike recoveries will be reported unless the dilution is more than 5X.

- 9.3.2. All surrogate, LCS, and MS recoveries (except for dilutions) must be entered into QuantIMS (when available) or other database so that accurate historical control limits can be generated. For tests without a separate extraction, surrogates and matrix spikes will be reported for all dilutions.

- 9.3.3. Refer to the QC Program document (QA-003) for further details of control limits.

9.4. Surrogates

All methods must use surrogates to the extent possible. Surrogate recoveries in samples and QC samples must be assessed to ensure that recoveries are within established limits. Surrogate recoveries must be met in the method blank (MB) and Laboratory Check Samples (LCS or LCS/LCSD). If any surrogates are outside limits, the following corrective actions must take place (except for dilutions):

- Check all calculations for error.
- Ensure that instrument performance is acceptable.
- Recalculate the data and/or reanalyze the extract if either of the above checks reveal a problem.
- The decision to reanalyze or flag the data should be made in consultation with the client. It is only necessary to reprepare / reanalyze 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: For DoD QSM and Ohio VAP Projects, all surrogates must meet criteria.

- 9.4.1. If dual column analysis is used the choice of which result to report is made in the same way as for samples (Section 12.1.2) unless one column is out of control, in which case the in-control result is reported.
- 9.4.2. 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 reparation is not necessary. If the sample is out of control and the MS and/or MSD is in control, then reparation or flagging of the data is required. Re-preparation includes the parent sample and MS/MSD.
- 9.4.3. Refer to the TestAmerica North Canton QC Program document (QA-003) for further details of the corrective actions.

9.5. Method Blanks

For each batch of samples, analyze a method blank. The method blank consists of reagent water for aqueous semivolatile samples, and sodium sulfate for semivolatile soils tests (Refer to SOP CORP-OP-0001NC for details). For low level volatiles, the method blank consists of reagent water. For medium level volatiles, the method blank consists of methanol as described in Appendix A. 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) or at or above 5% of the measured concentration of that analyte in the associated samples, whichever is higher.

If the analyte is a common laboratory contaminant (methylene chloride, acetone, 2-butanone, phthalate esters) 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-extraction and reanalysis 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. Such action should be taken in consultation with the client.

- 9.5.1. Refer to the TestAmerica North Canton QC Program document (QA-003) for further details of the corrective actions.
- 9.5.2. Refer to SOP NC-QA-0016 for further details concerning DoD Project Work.

9.6. Laboratory Control Samples (LCS)

For each batch of samples, analyze a LCS. The LCS contains a representative subset of the analytes of interest, and must contain the same analytes as the matrix spike. The LCS may also contain the full set of analytes with a subset of control analytes. If any control analyte or surrogate is outside established control limits, the system is out of control and corrective action must occur. Corrective action will normally be reparation and reanalysis of the batch.

- 9.6.1. Refer to the TestAmerica North Canton QC Program document (QA-003) for further details of the

corrective action.

9.6.2. If dual column analysis is used the choice of which result to report is made in the same way as for samples (Section 12.1.2) unless one column is out of control, in which case the in control result is reported.

9.6.3. LCS compound lists are included in the appendices.

9.7. Matrix Spikes

For each QC batch, analyze a matrix spike and matrix spike duplicate. Spiking compounds and levels are given in the appendices. Compare the percent recovery and relative percent difference (RPD) to those in the laboratory specific historically generated limits.

- 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.
- 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 reparation and reanalysis of the batch.
- If a MS/MSD is not possible due to limited sample, then a LCS duplicate should be analyzed. The recovery for each spike of the pair must be within established control limits. If the RPD is out of control but both accuracy recoveries are within acceptance criteria, prepare an NCM, and qualify report.
- The matrix spike / duplicate must be analyzed at the same dilution as the unspiked sample, unless the matrix spike components would then be above the calibration range.

9.7.1. If dual column analysis is used the choice of which result to report is made in the same way as for samples (Section 12.1.2) unless one column is out of control, in which case the in control result is reported.

10. CALIBRATION AND STANDARDIZATION

Internal or external calibration may be used. Internal calibration is recommended unless the sample matrix is likely to interfere with the quantitation of the internal standard. In either event prepare standards containing each analyte of interest at a minimum of five concentration levels. The low level standard must be at or below the reporting limit. The other standards define the working range of the detector. Recommended calibration levels are given in the appendices.

- 10.1. A new calibration curve must be generated after major changes to the system or when the continuing calibration criteria cannot be met. Major changes include new columns, changing PID lamps or FID jets or replacing the ECD detector. A new calibration is not required after clipping the column, replacing the septum or syringe, or other minor maintenance.
- 10.2. With the exception of Section 10.3 below, it is NOT acceptable to remove points from a calibration curve for the purpose of meeting criteria, unless the points are the highest or lowest on the curve AND the reporting limit and/or linear range is adjusted accordingly. In any event, at least five points must be included in the calibration curve. Quadratic (second order) calibrations require at least six points. Third order calibrations require at least seven points.
- 10.3. A level may be removed from the calibration if the reason can be clearly documented, for example a broken vial or no purge run. A minimum of five levels must remain in the calibration. The documentation must be retained with the initial calibration. Alternatively, if the analyst believes that a

point on the curve is inaccurate, the point may be reanalyzed and the reanalysis used for the calibration. All initial calibration points must be analyzed without any changes to instrument conditions, and all points must be analyzed within 24 hours.

10.4. External standard calibration

Quantitation by the external standard method assumes a proportional relationship between the calibration run and the analyte in the sample. To use this approach, introduce each calibration standard into the GC using the technique that will be used for samples. The ratio of the peak height or area response to the mass or concentration injected may be used to prepare a calibration curve.

$$\text{Calibration Factor (CF)} = \frac{\text{Area or Height of Peak}}{\text{Mass Injected (ng)}}$$

Some data systems may use the inverse of this formula. This is acceptable so long as the same formula is used for standards and samples. It is also possible to use the concentration of the standard rather than the mass injected. (This would require changes in the equations used to calculate the sample concentrations). Use of peak area or height must be consistent. However, if matrix interferences would make quantitation using peak area inaccurate for a particular sample, then peak height may be used as a substitute.

10.5. Internal standard calibration

10.5.1. The internal standard approach assumes that variations in instrument sensitivity, amount injected etc. can be corrected by determining the ratio of the response of the analyte to the response of an internal standard that has been added to the extract. To use this approach, select one or more internal standard(s) that are similar in analytical behavior to the compounds of interest. Recommended internal standards are given in the appendices. The analyst must demonstrate that the measurement of the internal standard is not affected by method or matrix interferences. If the sample matrix interferes with quantitation of the internal standard, then the external standard approach must be used instead. In this event use the response factors from the previous continuing calibration to quantitate the analytes in the sample with the interference (applies only to the sample with the interference).

10.5.2. Introduce each calibration standard into the GC using the technique that will be used for samples. Response factors (RF) for each compound are calculated as follows:

$$RF = \frac{A_s \times C_{is}}{A_{is} \times C_s}$$

Where:

A_s = Response for the analyte to be measured

A_{is} = Response for the internal standard

C_{is} = Concentration of internal standard

C_s = Concentration of the analyte to be determined in the standard

10.6. Calibration curve fits

Average response factor, linear regression, or quadratic curves may be used to fit the data. Average response factor may be used if the average % RSD of the response factors or calibration factors of all the analytes in the calibration standard taken together is $\leq 20\%$. The average %RSD is calculated by summing the RSD value for each analyte and dividing by the total number of analytes. NOTE: This is

note allowed for Ohio VAP Projects.

10.6.1. In general, for environmental analysis, average response factors are the most appropriate calibration model. Linear or curved regression fits should only be used if the analyst has reason to believe that the average RF model does not fit the normal concentration/response behavior of the detector.

10.6.2. Average response factor

The average response factor may be used if the average percent relative standard deviation (%RSD) of all the response factors taken together is $\leq 20\%$.

The equation for average response factor is:

$$\text{Average response factor} = \overline{RF} = \frac{\sum_{i=1}^n RF_i}{n}$$

Where: n = Number of calibration levels

$$\sum_{i=1}^n RF_i = \text{Sum of response factors for each calibration level}$$

10.6.3. Linear regression

The linear fit uses the following functions:

10.6.3.1. External Standard

$$y = ax + b$$

or

$$x = \frac{(y - b)}{a}$$

Where: y = Instrument response
 x = Concentration
 a = Slope
 b = Intercept

10.6.3.2. Internal Standard

$$C_s = \frac{\left[\frac{A_s C_{is}}{A_{is}} - b \right]}{a}$$

Where: C_s = Concentration in the sample

A_s = Area of target peak in the sample

A_{is} = Area of internal standard in the sample

C_{is} = Concentration of the internal standard

10.6.4. Quadratic curve

The quadratic curve uses the following functions:

10.6.4.1. External standard

$$y = ax + cx^2 + b$$

Where c is the curvature

10.6.4.2. Internal Standard

$$y = a \left(\frac{A_s \times C_{is}}{A_{is}} \right) + c \left(\frac{A_s \times C_{is}}{A_{is}} \right)^2 + b$$

10.7. Evaluation of calibration curves

10.7.1. The percent relative standard error (%RSE) from the calibration curve is used to evaluate the initial calibration. This provides a measure of how much error is associated with using the calibration curve for quantitation.

10.7.2. The least squares regression line is calculated and used to calculate the predicted concentration for each level. The percent relative standard error is calculated as follows:

$$\% RSE = 100\% \times \sqrt{\frac{\sum_{i=1}^N \left[\frac{C_i - PC_i}{C_i} \right]^2}{(N - P)}}$$

Where:

N = Number of points in the curve

P = Number of parameters in the curve (= 1 for average response factor, 2 for linear, 3 for quadratic)

C_i = True concentration for level i

PC_i = Predicted concentration for level i

Note that when average response factors are used, %RSE is equivalent to %RSD.

10.8. The following requirements must be met for any calibration to be used:

- Response must increase with increasing concentration.
- If a curve is used, the calculated intercept of the curve at zero response must be less than $\pm$ the reporting limit for the analyte.
- The average Relative Standard Error (RSD for average response factors) of the calibration points from the curve used must be $\leq 20\%$.
- Some data systems will not measure the %RSE from a linear or quadratic fit. For the linear case, the correlation coefficient may be used as an alternative to the %RSE, and must be greater than or equal to 0.990. For the quadratic case the Coefficient of Determination may be used, and must be greater or equal to 0.990.

Note: The Relative Standard Error (RSE) is superior to the Correlation Coefficient (r) and Coefficient of Determination (r^2) for testing the fit of a set of calibration points to a line. The lower points on a curve have little effect on r . As a result a curve may have a very good correlation coefficient (>0.995), while also having $> 100\%$ error at the low point.

10.9. Weighting of data points

10.9.1. In linear and quadratic calibration fits, the points at the lower end of the calibration curve have less absolute variance than points at the high concentration end of the curve. This can cause severe errors in quantitation at the low end of the calibration. 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 should be used if the data system has this capability.

10.10. Non-standard analytes are sometimes requested. For these analytes, it may be acceptable to analyze a single standard at the reporting limit with each continuing calibration rather than a five point initial calibration. This action must be with client approval. If the analyte is detected in any of the samples, a five point initial calibration must be generated and the sample(s) reanalyzed for quantitation.

10.11. Calibration Verification

10.11.1. 12-hour Calibration

10.11.1.1. The working calibration curve or RF must be verified by the analysis of a mid point calibration standard at the beginning, after every 12 hours, and at the end of the analysis sequence. The center of each retention time window is updated with each 12-hour calibration or calibration verification.

NOTE: This is not acceptable for Ohio VAP Projects.

10.11.2. Calibration Verification

10.11.2.1. It may be appropriate to analyze a mid point standard more frequently than every 12 hours. If these calibration verification standards are analyzed, requirements are the same as the 12-hour calibration with the exception that retention times are not updated.

10.11.3. Any individual compounds with $\%D < 15\%$ meet the calibration criteria. The calibration verification is also acceptable if the average of the $\%D$ for all the analytes is $< 15\%$. This average is calculated by summing the entire absolute $\%D$ results in the calibration (including

surrogates) and dividing by the number of analytes. Any analyte that is reportable as found must have a % difference of < 15% in the calibration verification or 12 hour calibration, on the column used for quantitation. Refer to section 12.1.2 for which result to report.

- 10.11.4. It is not necessary to run a calibration verification standard at the beginning of the sequence if samples are analyzed immediately after the completion of the initial calibration.
- 10.11.5. Samples quantitated by external standard methods must be bracketed by calibration verification standards that meet the criteria listed above. The bracketing standards on the column used for calibration must meet the same criteria as the opening standards. Bracketing is not necessary for internal standard methods.
- 10.11.6. If the analyst notes that a CCV has failed and can document the reason for failure (e.g. no purge, broken vial, carryover from the previous sample etc.) then a second CCV may be analyzed without any adjustments to the instrument. If this CCV meets criteria then the preceding samples have been successfully bracketed. If adjustments to the instrument are performed before the repeat CCV then the proceeding samples have not been successfully bracketed but analysis may continue.
- 10.11.7. In general, it is not advisable to analyze repeat CCVs on unattended runs. If repeat CCVs are analyzed then the first will serve as the bracketing standard for the preceding samples and the last will serve as the CCV for the following samples.
- 10.11.8. If highly contaminated samples are expected it is acceptable to analyze blanks or primers at any point in the run.
- 10.11.9. Percent difference calculation

10.11.9.1. Percent difference for internal and external methods is calculated as follows:

Internal Standard

External standard

$$\%D = \frac{RF_c - \overline{RF}}{\overline{RF}} \times 100$$

$$\%D = \frac{CF_c - \overline{CF}}{\overline{CF}} \times 100$$

Where: RF_c and CF_c are the response and calibration factors from the continuing calibration

$\overline{RF}$ and $\overline{CF}$ are the average response & calibration factors from the initial calibration

10.11.10. Percent drift calculation

- 10.11.10.1. Percent drift is used for comparing the continuing calibration to a linear or quadratic curve. The criteria for percent drift are the same as for percent difference

$$\% \text{ Drift} = \frac{\text{Calculated Conc.} - \text{Theoretical Conc.}}{\text{Theoretical Conc.}} \times 100\%$$

10.11.11. Corrective Actions for Continuing Calibration

- 10.11.11.1. If the overall average percent drift of all analytes is greater than $\pm 15\%$ corrective action must be taken. This may include clipping the column, changing the liner or other minor instrument adjustments, followed by reanalyzing the standard. If the overall average percent drift still varies by more than $\pm 15\%$, a new calibration curve must be prepared.

10.11.12. Corrective Action for Samples

- 10.11.12.1. For internal standard methods, any samples injected after a standard not meeting the calibration criteria must be re-injected.
- 10.11.12.2. For external standard methods, any samples injected after the last good continuing calibration standard must be re-injected.
- 10.11.12.3. If the average percent drift for all the analytes in the calibration is over 15%; but all of the analytes requested for a particular sample have percent drift $\leq 15\%$, then the analysis is acceptable for that sample.

11. PROCEDURE

11.1. Extraction

- 11.1.1. Extraction procedures are referenced in the SOP CORP-OP-0001NC, latest revision.

11.2. Cleanup

- 11.2.1. Cleanup procedures are referenced in the SOP NC-OP-0025, latest revision.

11.3. Gas Chromatography

- 11.3.1. Chromatographic conditions for individual methods are presented in the appendices.

11.4. Sample Introduction

- 11.4.1. In general, volatile analytes are introduced using purge and trap as described in Appendix A.
- 11.4.2. Semivolatile analytes are introduced by direct injection of the extract. Samples, standards, and QC must be introduced using the same procedure.

11.5. Analytical Sequence

- 11.5.1. An analytical sequence starts with an initial calibration or a calibration verification. Refer to the individual method appendices for method specific details of calibration verifications and analytical sequences.
- 11.5.2. The calibration verification includes analysis of standards containing all single response analytes and updating the retention time windows.
- 11.5.3. If there is a break in the analytical sequence of greater than 12 hours, a new analytical sequence must be started with a calibration verification.

11.6. Retention Time Windows

- 11.6.1. Retention time windows must be determined for all analytes. Make an injection of all analytes of interest each day over a three-day period. Calculate the standard deviation of the three

retention times for each analyte (relative retention times may also be used). For multi-response analytes (e.g., Aroclors) use the retention time of major peaks. Plus or minus three times the standard deviation of the retention times of each analyte defines the retention time window.

- 11.6.2. The center of the retention time window is the retention time from the last of the three standards. The centers of the windows are updated with the mid- point of the initial calibration and each 12-hour calibration. The widths of the windows will remain the same until new windows are generated following the installation of a new column.
- 11.6.3. If the retention time window as calculated above is less than ± 0.05 minutes, use a retention time window appropriate for the analysis and run time. This allows for slight variations in retention times caused by sample matrix.
- 11.6.4. The laboratory must calculate new retention time windows each time a new column is installed. The new windows must be generated within one week of the installation of the new column. Until these standards have been run on the new column, the retention time windows from the old column may be used, updated with the retention times from the new initial calibration.
- 11.6.5. Retention time studies are filed in the laboratory.
- 11.6.6. Corrective Action for Retention Times

- 11.6.6.1. The retention times of all compounds in the 12 hour calibration or calibration verification standard must be within the retention time window. If this condition is not met, all samples analyzed after the last compliant standard must be reanalyzed unless the following conditions are met for any compound that elutes outside the retention time window.

- 11.6.6.2. The retention time of that compound in the standard must be within a retention time range equal to twice the original window.

- 11.6.6.3. No peak that would be reportable may be present on the sample chromatogram within an elution time range equal to three times the original retention time window.

11.7. Daily Retention Time Windows

- 11.7.1. The center of the retention time windows determined in Section 11.6 are adjusted to the retention time of each analyte as determined in the 12 hour calibration standards or continuing calibration verification standards. (See the method 8081A and 8082 appendices for exceptions for multi-response components.) The retention time windows must be updated at the beginning of each analytical sequence and with each 12-hour calibration or continuing calibration verification.

11.8. Procedural Variations

- 11.8.1. Procedural variations are allowed only if deemed necessary in the professional judgment of the supervisor to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using a Nonconformance Memo and approved by a supervisor and QA/QC Manager. The Nonconformance Memo shall be filed in the project file. The nonconformance is also addressed in the case narrative. Any unauthorized deviations from this procedure must also be documented as a nonconformance, with a cause and corrective action described.

12. DATA ANALYSIS AND CALCULATIONS

12.1. Qualitative Identification

12.1.1. Tentative identification occurs when a peak is found within the retention time window for an analyte, at a concentration above the reporting limit, or above the MDL if J flags are required. Normally confirmation is required on a second column, but if the detector is sufficiently specific or if the sample matrix is well enough defined, single column analysis may be adequate. In some cases GC/MS confirmation may be required. Client specific requirements may also define the need for second column confirmation and/or GC/MS confirmation. Refer to the appendices for test specific requirements for confirmation. Identification is confirmed if a peak is also present in the retention time window for that analyte on the confirmatory column, at a concentration greater than the reporting limit (MDL if J flag confirmation required).

12.1.2. Dual column quantitation

For confirmed results, two approaches are available to the analyst:

- A) The primary column approach, or
- B) The better result approach

Both are acceptable to avoid the reporting of erroneous or unconfirmed data.

12.1.2.1. Primary column approach:

12.1.2.2. The result from the primary column is normally reported. The result from the secondary column is reported if any of the following three bulleted possibilities are true.

- There is obvious chromatographic interference on the primary column
- The result on the primary column is > 40% greater than the result on the secondary column
- Continuing or bracketing standard fails on the primary column but is acceptable on the secondary column. (If the primary column result is > 40% higher than the secondary and the primary column calibration fails, then the sample must be evaluated for reanalysis.)

12.1.2.3. Better result approach

The higher of the two results is normally reported. The higher result is considered better because the higher result is generally higher because of chromatographic interference. The lower result is reported if any of the following two bulleted possibilities are true.

- There is obvious chromatographic interference on the column with the higher result
- The continuing or bracketing calibration on the column with the higher result fails. (If the higher result is > 40% higher and the calibration on the column with the lower result fails, then the sample must be evaluated for reanalysis.)

12.1.3. If the Relative Percent Difference (RPD) between the response on the two columns is greater than 40%, or if the opinion of an experienced analyst is that the complexity of the matrix is resulting in false positives, the confirmation is suspect and the results are qualified. RPD is calculated using the following formula:

$$RPD = \frac{|R - R_2|}{\frac{1}{2}(R_1 + R_2)}$$

Where R=Result

12.1.4. Multi-response Analytes

For multi-response analytes, the analyst should use the retention time window, but should rely primarily on pattern recognition. The pattern of peaks will normally serve as confirmation.

12.1.5. The experience of the analyst should weigh heavily in the interpretation of the chromatogram. For example, sample matrix or laboratory temperature fluctuation may result in variation of retention times.

12.2. Calibration Range

12.2.1. If concentrations of any analytes exceed the working range as defined by the calibration standards, then the sample must be diluted and reanalyzed. Dilutions should 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.

12.3. Dilutions

12.3.1. 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 reanalyzed at a dilution targeted to bring the largest hit above 50% of the calibration range.

12.3.1.1. Guidance for Dilutions Due to Matrix

If the sample is initially run at a dilution and only minor matrix peaks are detected, then the sample should be reanalyzed at a more concentrated dilution. Analyst judgement is required to determine the most concentrated dilution that will not result in instrument contamination.

12.3.2. Reporting Dilutions

The most concentrated dilution with no target compounds above the calibration range will be reported. Other dilutions may be reported at client request if the lower dilutions will not cause detector saturation, column overload, or carryover. Analyst judgement and client site history will factors in the reporting of dual dilutions.

12.4. Interferences

12.4.1. If peak detection is prevented by interferences, further cleanup should be attempted. If no further cleanup is reasonable, then elevation of reporting levels and/or lack of positive identification must be addressed in the case narrative.

12.5. Internal Standard Criteria for Continuing Calibration

12.5.1. If internal standard calibration is used, then the internal standard response in a continuing calibration standard must be within 50 to 150% of the response in the mid level of the initial calibration.

12.6. Calculations

12.6.1. Capabilities of individual data systems may require the use of different formulas than those presented here. When this is the case, the calculations used must be shown to be equivalent and must be documented in an appendix attached to this document.

12.6.2. External Standard Calculations

12.6.2.1. Aqueous samples

$$\text{Concentration (mg / L)} = \frac{(A_x \times V_t \times D_f)}{(CF \times V_i \times V_s)}$$

Where:

A_x = Response for the analyte in the sample

V_i = Volume of extract injected, μL

D_f = Dilution factor

V_t = Volume of total extract, μL

V_s = Volume of sample extracted or purged, mL

CF = Calibration factor, area or height/ng, Section 10.1

12.6.2.2. Non-aqueous Samples

$$\text{Concentration (mg / kg)} = \frac{(A_x \times V_t \times D_f)}{(CF \times V_i \times W \times D)}$$

Where:

W = Weight of sample extracted or purged, g

$$D = \frac{100 - \% \text{ Moisture}}{100} \quad (D = 1 \text{ if wet weight is required})$$

12.6.3. Internal Standard Calculations

12.6.3.1. Aqueous Samples

$$\text{Concentration (mg / L)} = \frac{(A_x \times C_{is} \times D_f)}{(A_{is} \times RF \times V_s)}$$

Where:

C_{is} = Amount of internal standard added, ng

A_{is} = Response of the internal standard

RF = Response factor for analyte

12.6.3.2. Non-aqueous Samples

$$\text{Concentration (mg / kg)} = \frac{(A_x \times C_{is} \times D_f)}{(A_{is} \times RF \times W \times D)}$$

12.6.4. Surrogate Recovery

12.6.4.1. Concentrations of surrogate compounds are calculated using the same equations as for the target compounds. The response factor from the initial calibration is used. Surrogate recovery is calculated using the following equation:

$$\% \text{ Recovery} = \frac{\text{Concentration (or amount) found}}{\text{Concentration (or amount) spiked}} \times 100$$

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 SOPs NC-QA-0021 and S-Q-003.

13.2. Initial Demonstration

Each laboratory must make a one time initial demonstration of capability for each individual method. Demonstration of capability for both soils and water matrices is required. This requires analysis of QC check samples 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. Four aliquots of the QC check sample are analyzed using the same procedures used to analyze samples, including sample preparation. The concentration of the QC check sample should be equivalent to a mid-level calibration.

13.2.2. Calculate the average recovery and standard deviation of the recovery for each analyte of interest. Compare these results with the acceptance criteria given in each appendix.

13.2.3. If any analyte does not meet the 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 that an analyst who has been properly trained in its use and has the required experience performs this procedure.

14. POLLUTION PREVENTION

14.1. This method does not contain any specific modifications that serve to minimize or prevent pollution.

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 Safety Manual 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. **Vials containing sample extracts:** These vials are placed in the vial waste located in the GC/MS laboratory.
- 15.2.1.2. **Tubes containing sample extracts for TPH, Pesticides, PCBs and Herbicides:** These capped tubes are placed in the PCB/flammable waste located in the GC prep laboratory.
- 15.2.1.3. **Samples, standards, and all extraction materials contaminated with high levels (>50ppm) of PCB's 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. PCB containing samples are located through a LIMS query and disposed of as PCB containing.
- 15.2.1.4. **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.
- 15.2.1.5. **Discarded samples.** These samples are collected in the solid debris drum.

16. REFERENCES

- 16.1. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW846, 3rd Edition, Final Update III, December 1996, and Section 8000B
- 16.2. TestAmerica North Canton Quality Assurance Manual (QAM), current version
- 16.3. TestAmerica Corporate Safety Manual, M-E-0001, and TestAmerica North Canton Facility Addendum and Contingency Plan, current version
- 16.4. Associated SOPs and Policies, latest version
 - 16.4.1 QA Policy, QA-003
 - 16.4.2 Glassware Washing, NC-QA-0014
 - 16.4.3 Statistical Evaluation of Data and Development of Control Charts, NC-QA-0018
 - 16.4.4 Method Detection Limits and Instrument Detection Limits, CA-Q-S-006 and NC-QA-0021
 - 16.4.5 Standards and Reagents, NC-QA-0017
 - 16.4.6 Supplemental Practices for DoD Project Work, NC-QA-0016

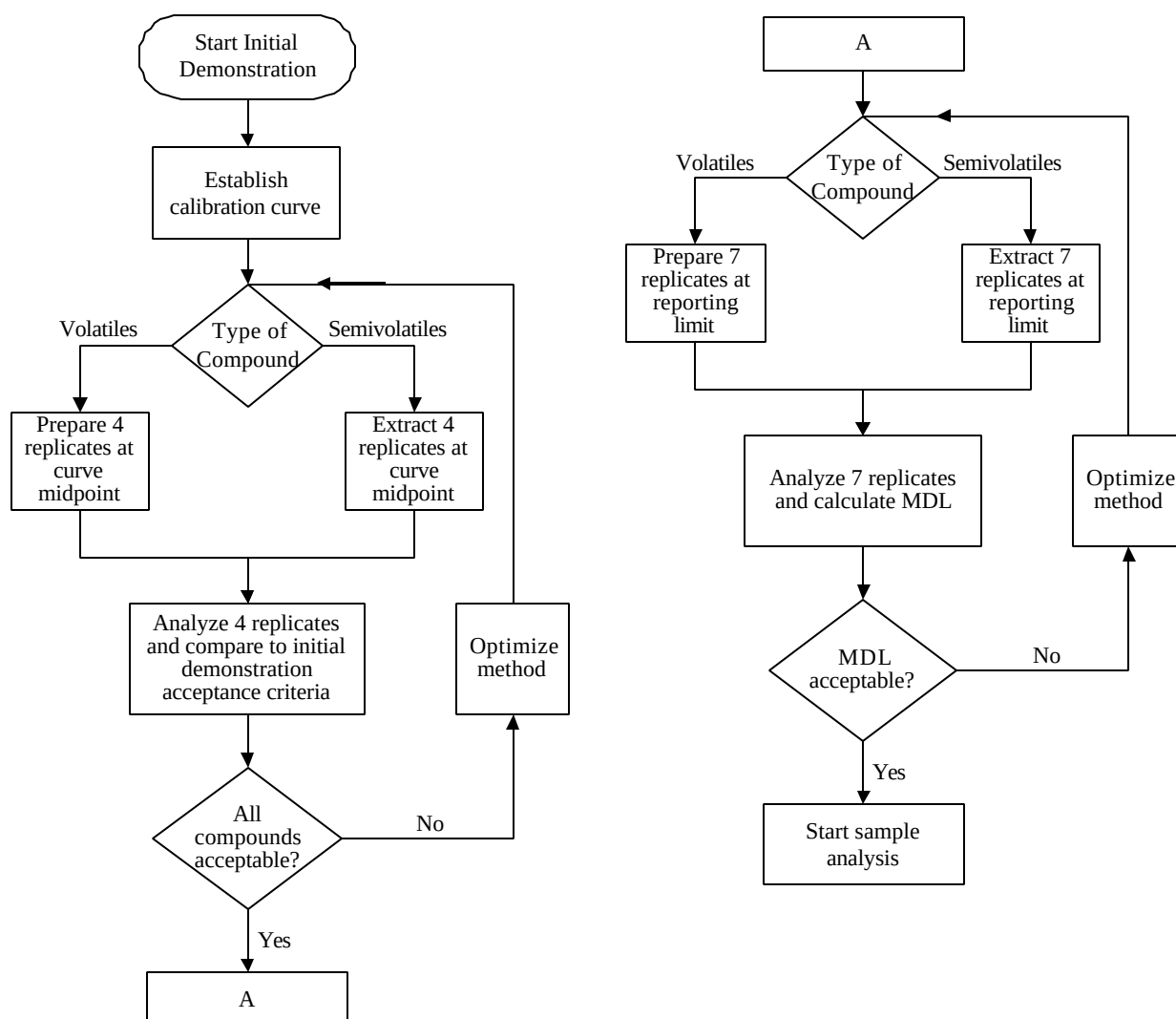
17. MISCELLANEOUS

- 17.1. Modifications from Reference Method

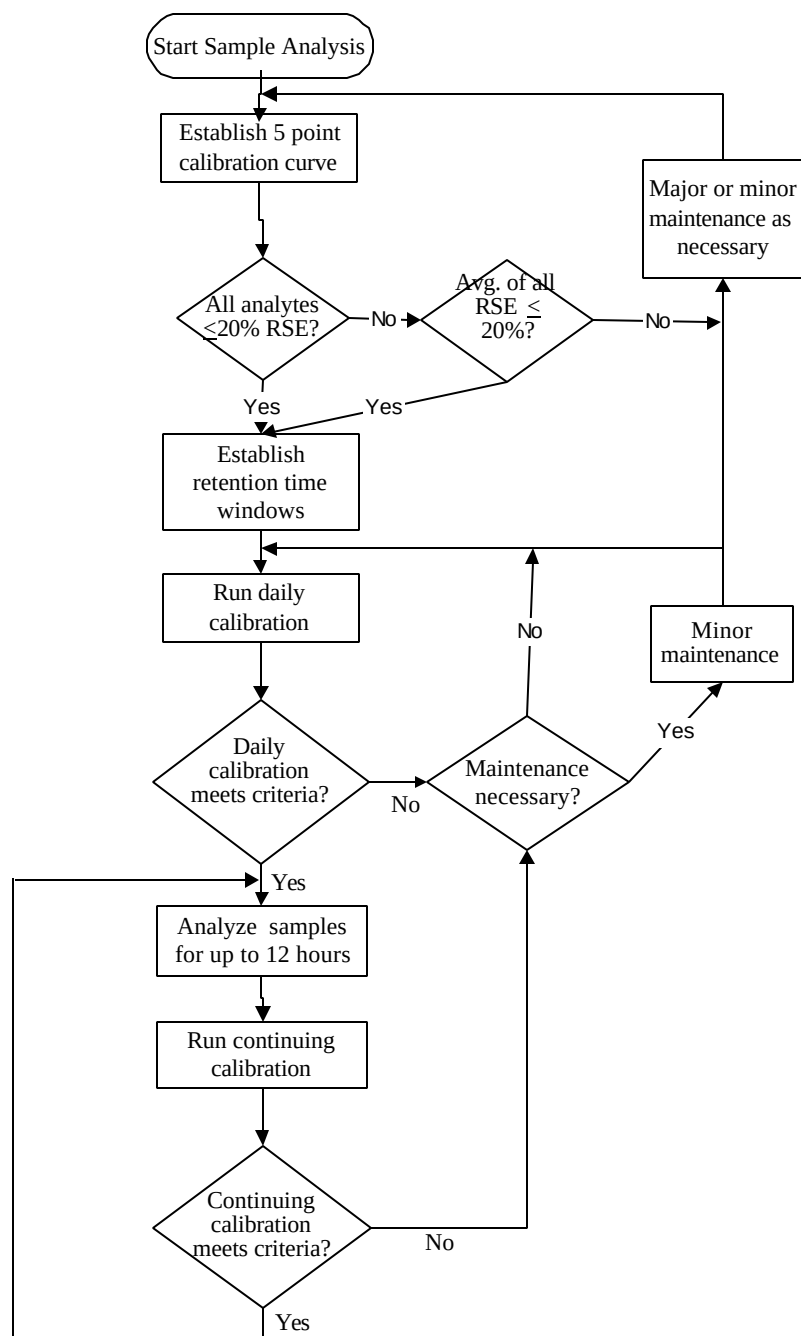
17.1.1. Chapter 1 of SW-846 states that the method blank should not contain any analyte of interest at or above the Method Detection Limit. This SOP states that the Method Blank must not contain any analyte of interest at or above the reporting limit. Common lab contaminants are allowed to be up to 5 times the reporting limit in the blank following consultation with the client.

17.2. Flow Diagrams

17.2.1. Initial demonstration and MDL



17.2.2 Sample Analysis¹



¹ This flow diagram is for guidance and cannot cover all eventualities. Consult the SOP text and a supervisor if in doubt.

1. SCOPE AND APPLICATION

- 1.1. This method describes sample preparation and extraction for the analysis of volatile organics by a purge and trap procedure following Method 8021B. All requirements of the 8000B section of this SOP must be met except when superseded by this Appendix. Refer to Table A-1 for the individual analytes normally determined by these procedures.
- 1.2. Compounds within the scope of this method have boiling points below 200°C and are insoluble or slightly soluble in water. Classes of compounds best suited to purge-and-trap analysis include low molecular weight halogenated hydrocarbons, aromatics, ketones, nitriles, acetates, acrylates, ethers, and sulfides.
- 1.3. Water samples and soils samples with low levels of contamination may be analyzed directly by purge-and-trap extraction and gas chromatography. Higher concentrations of these analytes in soil may be determined by the medium level methanol extraction procedure.
- 1.4. This method also describes the preparation of water-miscible liquids, non-water-miscible liquids, solids, wastes, and soils/sediments for analysis by the purge-and-trap procedure.
- 1.5. The associated LIMS method code is QR.

2. SUMMARY OF METHOD

- 2.1. An inert gas is bubbled through the sample at ambient temperature or at 40°C (40°C required for low-level soils), and the volatile components are transferred from the aqueous phase to the vapor phase. The vapor is swept through a sorbent column where the volatile components are adsorbed. After purging is completed, the sorbent column is heated and back-flushed with inert gas to desorb the components onto a gas chromatographic column. Analytes are detected using a photoionization Detector, an electrolytic conductivity detector or a combination of both.
- 2.2. For soil samples, a portion of the sample is dispersed in methanol to dissolve the volatile organic constituents. A portion of the methanolic solution is combined with water. It is then analyzed by purge-and-trap GC following the normal water method. If very low detection limits are needed for soil samples then direct purge using sodium bisulfate preservation may be necessary.

3. DEFINITIONS

- 3.1. Refer to the TestAmerica North Canton Laboratory Quality Manual (LQM), current version, for definitions of terms used in this SOP.

4. INTERFERENCES

- 4.1. Refer to Section 4 of the method 8000B part of this SOP for general information on chromatographic interferences.
- 4.2. Impurities in the purge gas, and from organic compounds out-gassing from the plumbing ahead of the trap, account for the majority of contamination problems. The analytical system must be demonstrated to be free from contamination under the conditions of the analysis by running laboratory reagent blanks. The use of non-TFE plastic tubing, non-TFE thread sealants, or flow controllers with rubber components in the purging device should be avoided.
- 4.3. Samples can be contaminated by diffusion of volatile organics (particularly methylene chloride and fluorocarbons) through the septum seal of the sample vial during shipment and storage. A trip blank

prepared from organic-free reagent water and carried through sampling and handling protocols serves as a check on such contamination.

- 4.4. Contamination by carryover can occur whenever high-concentration and low-concentration samples are analyzed sequentially. Whenever an unusually concentrated sample is analyzed, it should be followed by an analysis of organic-free reagent water to check for cross-contamination. The trap and other parts of the system are subject to contamination. Therefore, frequent bake-out and purging of the system may be required.
- 4.5. A holding blank is kept in the sample refrigerator. This is analyzed and replaced every 7 days. If the holding blank does not meet the method blank criteria, the source of contamination must be found and corrected. Evaluation of all samples analyzed in the 7-day period prior to the analysis of the contaminated holding blank is required.
- 4.6. Acidification of samples may result in hydrolysis of 2-chloroethyl-vinyl ether.

5. SAFETY

- 5.1. Refer to Section 5 of the Method 8000B section of this SOP for general safety requirements.
- 5.2. The following method analytes have been tentatively classified as known or suspected human or mammalian carcinogens: Benzene, Carbon Tetrachloride, 1,4-Dichlorobenzene, 1,2-Dichloroethane, Hexachlorobutadiene, 1,1,2,2-Tetrachloroethane, 1,1,2-Trichloroethane, Chloroform, 1,2-Dibromoethane, Tetrachloroethene, Trichloroethene, Vinyl Chloride. Pure standard materials and stock standard solutions of these compounds should be handled in a hood.
- 5.3. GC VOA instruments use an ultraviolet (UV) light source, which must be shielded from view.

6. EQUIPMENT AND SUPPLIES

- 6.1. Microsyringes -- 10µL, 25µL, 100µL, 250µL, 500µL, and 1000µL. These should be equipped with a 20 gauge (0.006" ID) needle. These will be used to measure and dispense methanolic solutions and aqueous samples.
- 6.2. Gas tight syringes -- 5 mL and 25 mL. Used for measuring sample volumes.
- 6.3. Purge and Trap Apparatus -- A device capable of extracting volatile compounds, trapping on a sorbent trap, and introducing onto a gas chromatograph.
- 6.4. Purge and Trap Autosampler -- In order to maintain high sample throughput, an autosampler is highly recommended.
- 6.5. Trap -- The trap used is dependent on the class of compound to be analyzed. Refer to Table A-2 for suggested traps for specific tests.
- 6.6. Purge Vessels -- These are dependent on the purge and trap unit/autosampler used. Both disposable culture tubes (needle sparge units) and specially designed vessels with fritted bottoms may be used. Follow the manufacturer's suggestions for configuration.
- 6.7. Columns - Refer to Table A-2 for details of columns.
- 6.8. Volumetric flasks, Class A: 5 mL to 250 mL
- 6.9. pH paper -- Range 0-14.

6.10. Balance capable of weighing to 0.01g for samples.

6.11. Chlorine Test Strips

6.12. Hach Chlorine Test Pillows

7. REAGENTS AND SUPPLIES

7.1. Refer to the method 8000B section of this SOP for general requirements for reagents and supplies.

7.2. Organic Free Water

7.2.1. Organic free water is defined as water in which an interferent is not observed at the reporting limit of the compounds of interest. The suggested method for generating organic free water is continuously sparging water with helium or nitrogen.

7.2.2. Other methods may be used, so long as the requirement that the water show no interference is met. The procedure used should be documented in a lab specific attachment.

7.3. Methanol -- Purge and Trap Grade

7.4. Standards

7.4.1. Refer to Tables A-5 and A-6 for details of surrogate, matrix spiking and internal standards. Calibration standard levels are not specified, since they may depend on the sensitivity and linear range of specific detectors. However, the low level standard must be equivalent to the reporting limits specified in Table A-1.

7.4.2. Volatile standards are prepared by injecting a measured volume of the stock standard into a syringe containing the appropriate volume of organic free water. The calibration standard is then loaded into the purge device.

8. SAMPLE COLLECTION, PRESERVATION, AND STORAGE

8.1. Holding times for all volatile analysis are 14 days from sample collection.

8.2. Water samples are normally preserved at pH < 2 with 1:1 hydrochloric acid. Unpreserved samples will be analyzed within seven days from sample collection.

8.3. Solid samples are field preserved 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. 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 5 g or 25 g 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 5 g EnCore samplers or to use field preservation.

8.5. Sample collection for medium level analysis using EnCore samplers.

8.5.1. Ship one 5 g (or 25 g) EnCore sampler per field sample position.

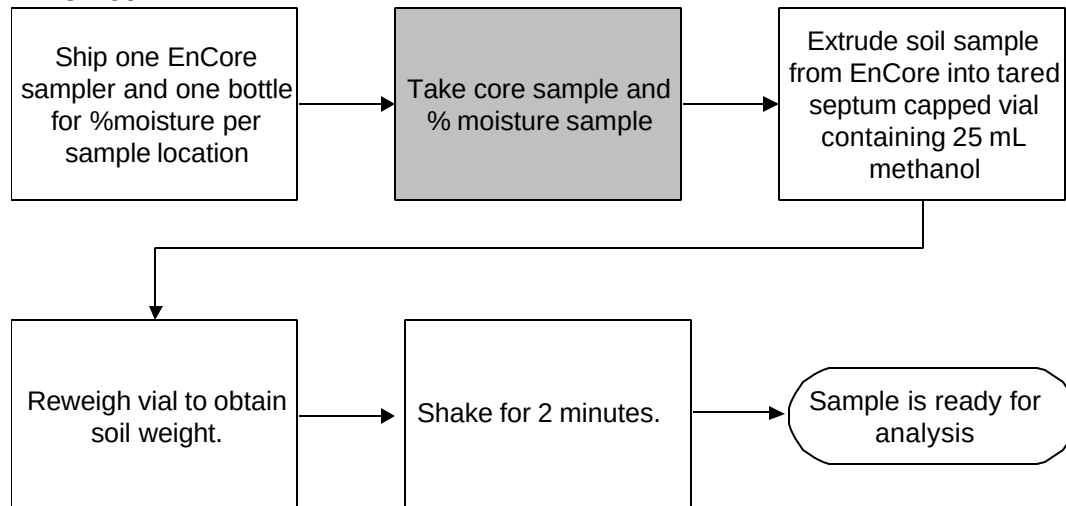
8.5.2. An additional bottle must be shipped for percent moisture determination.

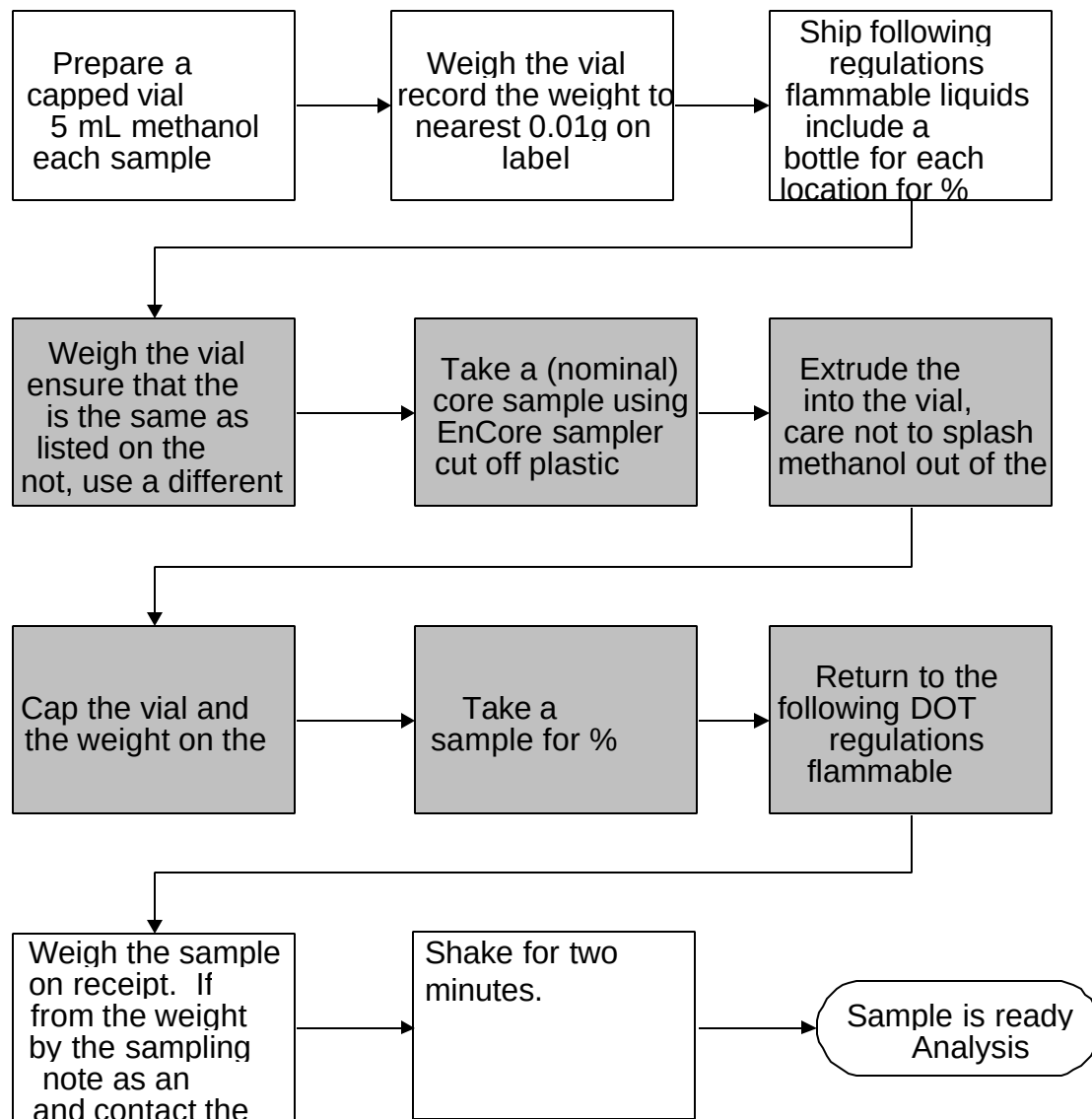
8.5.3. When the samples are returned to the lab, extrude the (nominal) 5g (or 25 g) sample into a tared

- VOA vial containing 5 mL methanol (25 mL methanol for the 25 g 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 100 µL of 250 µg/mL solution for a nominal 25 g sample, 20µL for a nominal 5 g sample.)
 - 8.5.5. Add the correct amount of matrix spiking solution to the matrix spike and matrix spike duplicate samples. (Add 100 µL of 250 µg/mL solution for a nominal 25 g sample, 20µL for a nominal 5 g sample.) The addition of spike introduces a slight error, (0.4%) which can be neglected, into the calculations.
 - 8.5.6. Prepare an LCS for each batch by adding the correct amount of matrix spiking solution to clean methanol. (100 µL of spike to 25 mL methanol or 20 µL spike to 5 mL methanol).
 - 8.5.7. Shake the samples for two minutes to distribute the methanol throughout the soil.
 - 8.5.8. Allow to settle and store in a clean Teflon capped vial at 4+2°C until analysis.
- 8.6. Sample collection for medium level analysis using field methanol preservation
- 8.6.1. Prepare a VOA vial by adding 5 mL purge and trap grade methanol. (If a 25 g sample is to be used, add 25 mL methanol to the 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 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 100 µL of 250 µg/mL solution for a nominal 25 g sample, 20µL for a nominal 5 g sample.)
 - 8.6.9. Add the correct amount of matrix spiking solution to the matrix spike and matrix spike duplicate samples. (Add 100 µL of 250 µg/mL solution for a nominal 25 g sample, 20µL for a nominal 5 g sample.) The addition of spike introduces a slight error, (0.4%) which can be neglected, into the calculations.
 - 8.6.10. Prepare an LCS for each batch by adding the correct amount of matrix spiking solution to clean methanol. (100 µL of spike to 25 mL methanol or 20 µL spike to 5 mL methanol).
 - 8.6.11. Shake the samples for two minutes to distribute the methanol throughout the soil.
 - 8.6.12. Allow to settle and store in a clean Teflon capped vial at 4+2°C until analysis.
- 8.7. Aqueous samples are stored in glass containers with Teflon lined septa at 4°C +/- 2°C, with minimum headspace.
- 8.8. Medium level solid extracts are aliquoted into 2 - 5 mL glass vials with Teflon lined caps and stored at

4°C +/- 2°C. The extracts are stored with minimum headspace.

- 8.9. 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 should 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.
- 8.10. A holding blank is stored with the samples. This is analyzed and replaced if any of the trip blanks show any contamination. Otherwise it is replaced every 14 days.

**EnCore procedure when low level is not required (field steps
in gray)**

Field methanol extraction procedure (field steps in gray)

9. QUALITY CONTROL

- 9.1. Refer to the method 8000B section of this SOP, section 9, for general quality control procedures, including batch definition, requirements for method blanks, LCS, matrix spikes, surrogates, and control limits.

10. CALIBRATION AND STANDARDIZATION

- 10.1. Refer to the Method 8000B section of this SOP, Section 10, for general calibration procedures.

10.2. Gas Chromatograph Operating Conditions

Various column configurations are possible. If dual column confirmation is necessary, the sample may be split using a Y splitter at the injector end to direct the sample to two columns and two detectors. For simultaneous determination of aromatic and halogenated volatiles, a single column is used and the PID and ELCD detectors are connected in series.

- 10.2.1. Refer to Table A-2, A-3 and A-4 for GC operating conditions. Additional operating instruction may be found in instrument manuals located in the laboratory.

10.3. Initial Calibration

- 10.3.1. Refer to Section 10 of the 8000B section of this SOP for details of initial calibration criteria.

- 10.3.2. Low-level soil samples must be purged at 40°C; therefore the calibration curve must also be purged at 40°C.

- 10.3.3. The low-level calibration must be at the reporting limit or below. The remaining standards encompass the working range of the detector.

- 10.3.4. Calibrate the instrument using the same volume that will be used during sample analysis.

10.4. Calibration Verification

- 10.4.1. A mid level calibration standard is used for the calibration verification. The gases have 20 % D criteria rather than the 15% used for other analytes. For analytes not listed in Method 8021B, the CCV criteria is 50% D.

- 10.4.2. A calibration verification run is performed after every ten samples for this method.

- 10.4.3. Bracketing of samples with calibration verification runs is only necessary for external standard analysis.

11. PROCEDURE

- 11.1. Refer to the method 8000B section of this SOP for general procedural requirements.

11.2. Analytical Sequence

- 11.2.1. The analytical sequence starts with an initial calibration of at least five points.

11.3. Confirmation

- 11.3.1. The PID and ELCD detectors are sufficiently selective that second column confirmation is not

always necessary. Requirements for second column confirmation should be decided in consultation with the client. If the PID and ELCD are used in series confirmatory information for many analytes can be gained by comparing the relative response from the two detectors.

The analytical sequence starts with an initial calibration of at least five points, or a 12 hour calibration that meets % difference criteria from an existing initial calibration.

11.4. Aqueous and Soil Sample Analysis (Purge and Trap units that sample directly from the VOA vial)

- 11.4.1. Check the pH of the sample remaining in the VOA vial prior to analysis. Samples are also checked for residual chlorine at this time.
- 11.4.2. Units, which sample from the VOA vial, should be equipped with a module, which automatically adds surrogate and internal standard solution, as needed, to the sample prior to purging the sample.
- 11.4.3. 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.
- 11.4.4. 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.

11.5. *Low-Level Solids Analysis using discrete autosamplers Bulk Solids*

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 sediment/soil sample mixed with reagent water containing the surrogate and, if applicable, internal and matrix spiking 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.

- 11.5.1. *Do not discard any supernatant liquids. Mix the contents of the container.*
- 11.5.2. *Weigh out 5 g (or other appropriate aliquot) of sample into a disposable culture tube or 40mL vial. Record the weight to the nearest 0.1 g. If method sensitivity is demonstrated, a smaller aliquot may be used. Do not use aliquots less than 0.5 g. If the sample is contaminated with analytes such that a purge amount less than 0.5g is appropriate, use the medium level method described in section 11.7.*
- 11.5.3. *Place in Autosampler.*
- 11.5.4. *Add 5 mL of organic free water to each vial. Add surrogate/internal standard (and matrix spike solutions if required.) (See Tables A-5, A-6, A-7 and A-8.) Add directly to the sample from 11.6.2.*
- 11.5.5. *The above steps should be performed rapidly and without interruption to avoid loss of volatile organics.*
- 11.5.6. *Soil samples that have low IS recovery when analyzed (<50%) should be reanalyzed once to confirm matrix effect. If external standard calibration is used, samples with surrogate recovery below the control limit should be reanalyzed once to confirm matrix effect.*

11.6. Methanol Extract Soils

- 11.6.1. 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 Section 8.5 or 8.6) to the syringe. Add internal standard (if used). Load the sample onto the purge and trap device and analyze as for aqueous samples. If less than 5µL of methanolic extract is to be added to the water, dilute the methanolic extract such that a volume greater than 5µL will be added to the water in the syringe.

12. DATA ANALYSIS AND CALCULATIONS

- 12.1. Refer to section 12 of the 8000B section of this SOP.

13. METHOD PERFORMANCE

- 13.1. Performance limits for the four replicate initial demonstration of capability required under Section 13.1 of the 8000B section of this SOP.

14. POLLUTION PREVENTION

- 14.1. This method does not contain any specific modifications that serve to minimize or prevent pollution.

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 Safety Manual 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 material from the auto-sampler.** Waste stream must be collected and neutralized before discharge to a sewer system if the pH is less than 4.
- 15.2.1.2. **Methanol waste from rinses and standards.** Methanol waste is discarded as a flammable liquid .
- 15.2.1.3. **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.

16. REFERENCES

- 16.1. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW846, 3rd Edition, Final Update III, December 1996, Sections 5000, 5030B, 5035 and 8021B.
- 16.2. 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.
- 16.3. Laboratory Holding Blanks, NC-QA-0020

17. MISCELLANEOUS

17.1. TABLES

Table A-1				
Standard Analyte List for Method 8021B				
Compound	CAS No.	Reporting Limit, µg/L or µg/kg		
		Aqueous	Low Soil	Medium Soil
1,1,1,2-Tetrachloroethane	630-20-6	1.0		
1,1,1-Trichloroethane	71-55-6	1.0		
1,1,2,2-Tetrachloroethane	79-34-5	1.0		
1,1,2-Trichloroethane	79-00-5	1.0		
1,1-Dichloroethane	75-34-3	1.0		
1,1-Dichloroethene	75-45-4	1.0		
1,1-Dichloropropene	563-58-6	1.0		
1,2,3-Trichlorobenzene	87-61-6	1.0		
1,2,3-Trichloropropane	96-18-4	1.0		
1,2,4-Trichlorobenzene	120-82-1	1.0		
1,2,4-Trimethylbenzene	95-63-6	1.0	1.0	50
1,2-Dibromo-3-Chloropropane(DBCP)	96-12-8	1.0		
1,2-Dibromoethane(EDB)	106-93-4	1.0		
1,2-Dichlorobenzene	95-50-1	1.0		
1,2-Dichloroethane	107-06-2	1.0		
1,2-Dichloropropane	78-87-5	1.0		
1,3,5-Trimethylbenzene	108-67-8	1.0	1.0	50
1,3-Dichlorobenzene	541-73-1	1.0	1.0	50
1,3-Dichloropropane	142-28-9	1.0		
1,4-Dichlorobenzene	106-46-7	1.0	1.0	50
2,2-Dichloropropane	590-20-7	1.0		
2-Chloroethyl vinyl ether	110-75-8	5.0		
2-Chlorotoluene	95-49-8	1.0		
4-Chlorotoluene	106-43-4	1.0		
Acetone	67-64-1	10		
Benzene	71-43-2	1.0	1.0	50
Benzyl Chloride	100-44-7	5.0		
Bromobenzene	108-86-1	1.0		
Bromochloromethane	74-97-5	1.0		
Bromodichloromethane	75-27-4	1.0		
Bromoform	75-25-2	1.0		
Bromomethane	74-83-9	1.0		
Carbon Tetrachloride	56-23-5	1.0		
Chlorobenzene	108-90-7	1.0		

Table A-1				
Standard Analyte List for Method 8021B				
Compound	CAS No.	Reporting Limit, µg/L or µg/kg		
		Aqueous	Low Soil	Medium Soil
Chlorodibromomethane	124-48-1	1.0		
Chloroethane	70-00-3	1.0		
Chloroform	67-66-3	1.0		
Chloromethane	74-87-3	1.0		
cis-1,2 Dichloroethene	156-59-4	1.0		
cis-1,3-Dichloropropene	10061-01-5	1.0		
Dibromomethane	74-95-3	1.0		
Dichlorodifluoromethane	75-71-8	1.0		
Ethyl Benzene	100-41-4	1.0	1.0	50
Freon 113	76-13-1	1.0		
Hexachlorobutadiene	87-68-3	1.0		
Isopropylbenzene	98-82-8	1.0		
MEK (2-butanone)	78-93-3	5.0		
Methyl tert-butyl ether (MTBE)	1634-04-4	1.0		
Methylene Chloride	75-09-2	5.0		
MIBK (4-methyl-2-pentanone)	108-10-1	5.0		
Naphthalene	91-20-3	2.0	2.0	250
n-butylbenzene	104-51-8	1.0		
n-Propylbenzene	10306501	1.0		
p-Isopropyltoluene	99-87-6	1.0		
sec-Butylbenzene	135-98-8	1.0		
Styrene	100-42-5	1.0		
tert-Butylbenzene	98-06-6	1.0		
Tetrachloroethene	127-18-4	1.0		
Toluene	108-88-3	1.0	1.0	50
trans-1,2-Dichloroethene	156-60-5	1.0		
trans-1,3-Dichloropropene	10061-02-6	1.0		
Trichloroethene	79-01-6	1.0		
Trichlorofluoromethane	75-69-4	1.0		
Vinyl Chloride	75-01-4	1.0		
Xylenes (total)	1330-20-7	1.0	1.0	50

Table A-2 Recommended Conditions for Method Combined Aromatic and Halogenated Volatiles	
Parameter	Recommended Conditions
Temperature program	35°C, 12 min, then 4°C/min to 200°C, hold for 5 min
Column 1	DB-VRX or RTX-502.2 105m x 0.53 mm id df = 3.0um
Column 2	DB-1 or RTX-1 105m x 0.53 mm ID df = 3.0um
Column 3	Rtx - Volatiles 120m x 0.53mm ID df=2.0um
Carrier gas	Helium
Purge Flow / time	40 mL/min, 11 minutes
Desorb Temp / time	180°C, 2 minutes (220°C for Vocarb 3000)
Bake Time / temp	200°C, 12 minutes (230°C for Vocarb 3000)
Transfer line / valve temp	115°C

Table A-3 Surrogate and Internal Standard Concentrations				
Standard	Components	Working Solution µg/mL	Spike amount µL (for 5 mL purge)	Final concentration µg/L (µg/kg)
Combined aromatic and halogenated volatiles IS/SS	Fluorobenzene (SS)	50	1	10
	1,4-Dichlorobutane (SS)	50	1	10
	a,a,a-Trifluorotoluene	50	1	10

It may be necessary to select different surrogates in order to minimize sample interferences. 1-chloro-4-fluorobenzene and 4-chlorotoluene are fairly well resolved from analytes listed in this SOP. However 4-chlorotoluene may sometimes be requested as a target analyte. Other surrogates that may be considered, and issues associated with their use are:

Bromochloromethane:	Elutes very close to chloroform and cis-1, 2-dichloroethene on the 502.2 column. May be a target analyte.
1-Chloro-2-fluorobenzene:	Elutes close to ethylbenzene on DB-1 or Rtx-1 and close to m,p-xylene on 502.2
Bromofluorobenzene:	Close to 1,1,2,2-trichloroethane and 1,2,3-trichloropropane on the 502.2 column. Good on DB-1 or Rtx-1.
2-Bromo-1-chloropropane:	May coelute with 1,1,2-trichloroethane

Table A-4				
Concentrations for LCS and MS/MSD compounds				
Standard	Components	Working Solution µg/mL	Spike amount µL (5 mL purge)	Final concentration µg/L (µg/kg)
Aromatic	Benzene	20	5	20
	Toluene	20		20
	Chlorobenzene	20		20
Halogenated	Chlorobenzene	20	5	20
	1,1-Dichloroethene	20		20
	Trichloroethene	20		20
Combination aromatic / Halogenated	Benzene	20	5	20
	Toluene	20		20
	Chlorobenzene	20		20
	1,1-Dichloroethene	20		20
	Trichloroethene	20		20

1. SCOPE AND APPLICATION

- 1.1. This SOP Appendix describes procedures to be used when SW-846 Method 8081A is applied to the analysis of organochlorine pesticides by GC/ECD. This Appendix may also be applied when discontinued SW-846 Method 8080A is requested, and is applicable to extracts derived from any matrix which are prepared according to the appropriate TestAmerica North Canton sample extraction SOPs. (CORP-OP-0001NC)
- 1.2. Table B-1 lists compounds, which are routinely determined by this method, and gives the Reporting Limits (RL) for each matrix. RLs given are based on the low level standard and the sample preparation concentration factors. Matrix interferences may result in higher RLs than those listed.
- 1.3. At client request, this method may also be used for the analysis of PCBs (Aroclors) in combination with pesticides, although these are normally analyzed following method 8082, as described in Appendix C of this SOP. Extracts that have been acid cleaned may not be analyzed for pesticides, since several of the pesticides will be degraded.
- 1.4. The associated LIMS method code is QJ (8081A).

2. SUMMARY OF METHOD

- 2.1. This method presents conditions for the analysis of prepared extracts of organochlorine pesticides. The pesticides are injected onto the column and separated and detected by electron capture detection. Quantitation is by internal or external standard methods.

3. DEFINITIONS

- 3.1. Refer to the TESTAMERICA North Canton Laboratory Quality Manual (LQM), current version, for definitions of terms used in this document.

4. INTERFERENCES

- 4.1. Refer to the method 8000B section of this SOP for information regarding chromatographic interferences.
- 4.2. Interferences in the GC analysis arise from many compounds amenable to gas chromatography that give a measurable response on the electron capture detector. Phthalate esters, which are common plasticizers, can pose a major problem in the determinations. Avoiding contact with any plastic materials minimizes interferences from phthalates.
- 4.3. Sulfur will interfere and can be removed using procedures described in SOP NC-OP-0025, Cleanup SOP.
- 4.4. Interferences co-extracted from samples will vary considerably from source to source. The presence of interferences may raise quantitation limits for individual samples. Using hexane / acetone as the extraction solvent (rather than hexane / methylene chloride) will reduce the amount of interferences extracted.

5. SAFETY

- 5.1. Refer to Section 5 of the Method 8000B SOP for general safety requirements.
- 5.2. Aroclors have been classified as a potential carcinogen under OSHA. Concentrated solutions of Aroclors must be handled with extreme care to avoid excess exposure. Contaminated gloves and clothing must be removed immediately. Contaminated skin surfaces must be washed thoroughly.

- 5.3. The following parameters covered by this method have been tentatively classified as known or suspected, human or mammalian carcinogens: 4,4'-DDT, 4,4'-DDD, and the BHCs. Primary standards of these toxic compounds should be prepared in a hood.
- 5.4. All ^{63}Ni sources shall be leak tested every six months, or in accordance with the manufacturer's general radioactive material license.
- 5.5. All ^{63}Ni sources shall be inventoried every six months. If a detector is missing, the Director, EH&S shall be immediately notified and a letter sent to the NRC or local state agency.

6. EQUIPMENT AND SUPPLIES

- 6.1. Refer to Section 6 of the 8000B section of this SOP. A ^{63}Ni electron capture detector is required.
- 6.2. Refer to Table B-2 for analytical columns.
- 6.3. Microsyringes, various sizes, for standards preparation, sample injection, and extract dilution.

7. REAGENTS AND STANDARDS

- 7.1. Refer to the method 8000B section of this SOP for general requirements for reagents and supplies.
- 7.2. Refer to Table B-3 for details of calibration standards.
- 7.3. Surrogate Standards
 - 7.3.1. Tetrachloro-m-xylene and decachlorobiphenyl are the surrogate standards. Refer to tables B-5 and B-6 for details of surrogate standards.
- 7.4. Column Degradation Evaluation Mix
 - 7.4.1. A mid-level standard containing 4,4'-DDT and Endrin and not containing any of their breakdown products must be prepared for evaluation of degradation of these compounds by the GC column and injection port. This mix must be replaced after one year, or whenever corrective action to columns fails to eliminate the breakdown of the compounds, whichever is shorter. This solution also contains the surrogates. Refer to Table B-4 for details of the column degradation evaluation mix.

8. SAMPLE COLLECTION, PRESERVATION AND STORAGE

- 8.1. Refer to Section 8 of the 8000B section of this SOP.

9. QUALITY CONTROL

- 9.1. Refer to Section 9 of the 8000B section of this SOP.

10. CALIBRATION AND STANDARDIZATION

- 10.1. Refer to Section 10 of the 8000B section of this SOP for general calibration requirements.
- 10.2. Refer to Table B-2 for recommended details of GC operating conditions. The conditions listed should result in resolution of all analytes listed in Table B-1 on both columns.
- 10.3. Column Degradation Evaluation

- 10.3.1. Before any calibration runs, either initial or 12 hour, The column evaluation mix must be injected before each initial or daily calibration. The degradation of DDT and endrin must be calculated (see equations 9 and 10) and each shown to be less than 15% before calibration can proceed. This is only necessary if the target compound list includes DDT, Endrin, or any of their degradation products.
- 10.3.2. If the breakdown of DDT and/or endrin exceeds the limits given above, corrective action must be taken. This action may include:
- 10.4. Replacement of the injection port liner or the glass wool.
- 10.5. Cutting off a portion of the injection end of a capillary column.
- 10.6. Replacing the GC column.
- 10.7. Initial Calibration
 - 10.7.1. Refer to Section 10 of the 8000B section of this SOP for details of calibration procedures.
 - 10.7.2. Refer to Table B-7 for the initial calibration analytical sequence.
 - 10.7.3. The response for each single-peak analyte will be calculated by the procedures described in the general method for GC analysis.
 - 10.7.4. The surrogate calibration curve is calculated from the Individual AB mix. Surrogates in the other calibration standards are used only as retention time markers. If there are resolution problems, then the A and B mixes may be analyzed separately.
 - 10.7.5. For multi-component pesticides:
 - 10.7.5.1. A five-point calibration is used for multi-component pesticides (typically toxaphene and technical chlordane). Two options are possible; the same quantitation option must be used for standards and samples. Refer to section 12.3 for guidance on which option to use.
 - 10.7.6. A full 5 point calibration for any of the multi-component analytes is analyzed.
- 10.8. 12-hour Calibration Verification
 - 10.8.1. The 12-hour calibration verification sequence must be analyzed within 12 hours of the start of the initial calibration and at least once every 12 hours thereafter if samples are being analyzed. If more than 12 hours have elapsed since the injection of the last sample in the analytical sequence, a new analytical sequence must be started with a 12 hour calibration. A mid level calibration standard is used for the 12 hour calibration. Refer to the 8000B section of this SOP for acceptance criteria.
 - 10.8.2. At a minimum, the 12-hour calibration includes analysis of the breakdown mix followed by mid level standards of any single and multi-component analytes.
 - 10.8.3. The retention time windows for any analytes included in the 12-hour calibration are updated.
- 10.9. Continuing Calibration

- 10.9.1. The AB calibration mix is analyzed as the continuing calibration standard. At a minimum, this is analyzed after every 20 samples, including matrix spikes, LCS, and method blanks. If 12 hours elapse analyze the 12 hour standard sequence instead. The continuing calibration standard need not include multi-component analytes. If instrument drift is expected due to sample matrix or other factors, it may be advisable to analyze the continuing calibration standard more frequently.
- 10.9.2. A mid level calibration standard is used for the continuing calibration.

11. PROCEDURE

- 11.1. Refer to the method 8000B section of this SOP for general procedural requirements.
- 11.2. Extraction
 - 11.2.1. The extraction procedure is described in SOP No. CORP-OP-0001NC.
- 11.3. Cleanup
 - 11.3.1. Cleanup procedures are described in SOP No. NC-OP-0025.
- 11.4. Suggested gas chromatographic conditions are given in Table B-2.
- 11.5. Allow extracts to warm to ambient temperature before injection.
- 11.6. The suggested analytical sequence is given in Table B-7.

12. DATA ANALYSIS AND CALCULATIONS

- 12.1. Refer to the 8000B section of this SOP for identification and quantitation of single component analytes.
- 12.2. Identification of Multi-component Analytes
 - 12.2.1. Retention time windows are also used for identification of multi-component analytes, but the “fingerprint” produced by major peaks of those compounds in the standard is used in tandem with the retention times to identify the compounds. The ratios of the areas of the major peaks are also taken into consideration. Identification of these compounds may be made even if the retention times of the peaks in the sample fall outside of the retention time windows of the standard, if in the analyst’s judgment the fingerprint (retention time and peak ratios) resembles the standard chromatogram.
- 12.3. Quantitation of Multi-component Analytes
 - 12.3.1. Use 3-10 major peaks (or total area for quantitation) as described in section 10.4.4, initial calibration of multi-component analytes.
 - 12.3.2. If there are no interfering peaks within the envelope of the multi-component analyte, the total area of the standards and samples may be used for quantitation. Any surrogate or extraneous peaks within the envelope must be subtracted from the total area.
 - 12.3.2.1. Multiple peak option
 - 12.3.3. This option is particularly valuable if toxaphene is identified but interferences make quantitation based on total area difficult. Select 3-10 major peaks in the analyte pattern. Calculate the response using the total area or total height of these peaks. Alternatively, find

the response of each of the 3-10 peaks per multi-peak pesticide, and use these responses independently, averaging the resultant concentrations found in samples for a final concentration result. When using this option, it is appropriate to remove peaks that appear to be coeluting with contaminant peaks from the quantitation. (i.e. peaks which are significantly larger than would be expected from the rest of the pattern.)

- 12.3.4. Chlordane may be quantitated either using the multiple peak option (12.3.1.1) total area option (12.3.1.2.) or by quantitation of the major components, α -chlordane, γ -chlordane and heptachlor.

12.4. Total area option

- 12.4.1. The total area of the standards and samples may be used for quantitation of multi-component analytes. Any surrogate or extraneous peaks within the envelope must be subtracted from the total area. This option should not be used if there are significant interference peaks within the multi-component pattern in the samples. The retention time window for total area measurement must contain at least 90% of the area of the analyte.

- 12.5. Second column confirmation multi-component analytes will only be performed when requested by the client, because the appearance of the multiple peaks in the sample usually serves as a confirmation of analyte presence.

- 12.6. Surrogate recovery results are calculated and reported for decachlorobiphenyl (DCB) and tetrachloro-m-xylene (TCMX). Corrective action is only necessary if DCB and TCMX are both outside of acceptance limits.

12.7. Calculation of Column Degradation/% Breakdown (%B)

Equation 9

$$12.7.1. \text{ DDT \%B} = \frac{A_{DDD} + A_{DDE}}{A_{DDD} + A_{DDE} + A_{DDT}} \times 100$$

Where:

A_{DDD} , A_{DDE} , and A_{DDT} = the response of the peaks for 4,4'-DDD, 4,4'-DDE, and 4,4'-DDT in the column degradation evaluation mix.

Equation 10

$$\text{Endrin \%B} = \frac{A_{EK} + A_{EA}}{A_{EK} + A_{EA} + A_E} \times 100$$

Where:

A_{EK} , A_{EA} , and A_E = the response of endrin ketone, endrin aldehyde, and endrin in the column degradation evaluation mix.

13. METHOD PERFORMANCE

- 13.1. Performance limits for the four replicate initial demonstration of capability required under Section 13.1 of the main body of this SOP. The spiking level should be equivalent to a mid level calibration.

14. POLLUTION PREVENTION

- 14.1 Refer to Section 14 of the 8000B section of this SOP.

15. WASTE MANAGEMENT

- 15.1. Waste generated in this procedure will be segregated and disposed according to the facility hazardous waste procedures. The Environmental Health and Safety Director should be contacted if additional information is required.

16. REFERENCES

- 16.1. SW846, Update III, December 1996, Method 8081A

17. MISCELLANEOUS

- 17.1. Modifications from Reference Method - None
17.2. TABLES

Table B-1			
Standard Analyte List and Reporting Limits for Method 8081A			
Compound	Reporting Limit, µg/L or µg/kg		
	water	soil	waste
Aldrin	0.05	1.7	50
α-BHC	0.05	1.7	50
β-BHC	0.05	1.7	50
δ-BHC	0.05	1.7	50
γ-BHC (Lindane)	0.05	1.7	50
α-Chlordane	0.05	1.7	50
γ-Chlordane	0.05	1.7	50
Chlordane (technical)	0.5	17	500
4,4'-DDD	0.05	1.7	50
4,4'-DDE	0.05	1.7	50
4,4'-DDT	0.05	1.7	50
Dieldrin	0.05	1.7	50
Endosulfan I	0.05	1.7	50
Endosulfan II	0.05	1.7	50
Endosulfan Sulfate	0.05	1.7	50
Endrin	0.05	1.7	50
Endrin Aldehyde	0.05	1.7	50
Heptachlor	0.05	1.7	50
Heptachlor Epoxide	0.05	1.7	50
Methoxychlor	0.1	3.3	100
Toxaphene	2.0	67	2000
APPENDIX IX ADD-ONS			
Diallate	1.0	33	1000
Isodrin	0.1	3.3	100
Chlorobenzilate	0.1	3.3	100
Kepone ¹	1.0	33	1000

¹ Kepone is sometimes requested for analysis by method 8081A. However kepone may produce peaks with broad tails that elute later than the standard by up to a minute (presumably due to hemi-acetal formation). As a result kepone analysis by 8081A is unreliable and not recommended. Analysis by method 8270C is a possible alternative. Note: alpha chlordane, gamma chlordane, and endrin ketone are not required for some projects. The following concentration factors are assumed in calculating the Reporting Limits:

APPENDIX B

SOP No. NC-GC-038, Rev. 0
Effective Date: 01/21/08
Page B7 of B11

	<u>Extraction Vol.</u>	<u>Final Vol.</u>
Ground water	1000 mL	10 mL
Low-level Soil	30 g	10 mL
High-level soil / waste	1 g	10 mL

Table B-2 Recommended Conditions for Method 8081A	
Parameter	Recommended Conditions
Injection port temp	220°C
Detector temp	325°C
Temperature program	120°C for 1 min, 8.5°C/min to 285°C, , 6 min hold
Column 1	Rtx-CLPesticides 30m x 0.32mm id, 0.5µm
Column 2	Rtx-35 30m x 0.32 mm id, 0.5µm
Column 3	DB-608, 30m X 0.32 mm, 0.25µm
Injection	2µL
Carrier gas	Helium or Hydrogen
Make up gas	Nitrogen
Y splitter	Restek or J&W or Supelco glass tee

Table B-3 Calibration Levels ng/mL for Method 8081A						
	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6 ²
Individual Mix AB¹						
Aldrin	5	10	25	50	100	200
g-BHC (Lindane)	5	10	25	50	100	200
Heptachlor	5	10	25	50	100	200
Methoxychlor	10	20	50	100	200	400
Dieldrin	5	10	25	50	100	200
Endosulfan I	5	10	25	50	100	200
Endosulfan II	5	10	25	50	100	200
4,4'-DDT	5	10	25	50	100	200
Endrin Aldehyde	5	10	25	50	100	200
Endrin Ketone	5	10	25	50	100	200
β-BHC	5	10	25	50	100	200
δ-BHC	5	10	25	50	100	200
α-BHC	5	10	25	50	100	200
4,4'-DDD	5	10	25	50	100	200
4,4'-DDE	5	10	25	50	100	200
Endosulfan Sulfate	5	10	25	50	100	200
Endrin	5	10	25	50	100	200
α-Chlordane ³	5	10	25	50	100	200
γ-Chlordane ³	5	10	25	50	100	200
Multi-component Standards						
Chlordane (Technical)	20	50	100	200	500	
Toxaphene	200	500	1000 ⁵	2000	5000	
Surrogates are included with all the calibration mixes at the following levels:						
Tetrachloro-m-xylene	5	10	25	50	100	200
Decachlorobiphenyl	5	10	25	50	100	200

¹ Standards may be split into an A and B mix if resolution of all compounds on both columns is not obtained.² Level 6 is optional and should only be used if linearity can be maintained on the instrument to this level.³ Compounds may be used in lieu of running a daily technical Chlordane standard for samples that are non-detect for technical Chlordane.

Table B-4	
Column Degradation Evaluation Mix ng/mL for Method 8081A	
Component	Concentration
4,4'-DDT	25
Endrin	25
Tetrachloro-m-xylene (Surrogate)	20
Decachlorobiphenyl (Surrogate)	20

Table B-5			
LCS/Matrix Spike and Surrogate Spike levels mg/L or mg/kg for Method 8081A			
	Aqueous	Soil	Waste
gamma BHC (Lindane)	0.20	33.3	200
Aldrin	0.20	33.3	200
Heptachlor	0.20	33.3	200
Dieldrin	0.50	33.3	500
Endrin	0.50	33.3	500
4,4'-DDT	0.50	33.3	500
Tetrachloro-m-xylene (Surrogate)	0.20	33.3	200
Decachlorobiphenyl (Surrogate)	0.20	33.3	200

Table B-6		
LCS/Matrix Spike and Surrogate Spike levels for TCLP mg/L or mg/kg for Method 8081A		
	Aqueous	Waste
Heptachlor	5	500
Heptachlor epoxide	5	500
Lindane	5	500
Endrin	5	500
Methoxychlor	10	1000

Table B-7
Suggested Analytical Sequence for Method 8081A

Initial Calibration

Solvent blank (optional)	
Primer if needed	
Breakdown Mix	
Individual mix AB	All levels
Technical Chlordane	Level 3 ¹
Toxaphene	Level 3 ¹
Up to 20 samples unless 12 hours comes first)	
Solvent blank (optional)	
Individual mix AB	Mid level (Continuing calibration)
Samples	
After 12 hours:	
Breakdown mix	
Individual mix AB	
Any other single component analytes	
Any multi-component analytes	

¹ A five-point curve for any of the multi-component analytes may be included

If Aroclors are included, a 5 point calibration for Aroclor 1016/1260 should be included with the initial calibration and a single point for the other Aroclors. The mid point 1016/1260 mix is included with the daily calibration (every 12 hours).

Note: A solvent blank or primer may be analyzed at any time during the sequence when highly contaminated samples are expected. A solvent blank or primer may not be analyzed as routine immediately prior to standards.

Note: The initial primer is used if the instrument has been idle for a period of time.

12 -Hour Calibration

At least every 12 hours, counting from the start of the initial calibration, or from the start of the last daily calibration, the retention time windows must be updated using the Individual mix AB, and the breakdown mix must be run before the continuing calibration.

1. SCOPE AND APPLICATION

1.1. This SOP Appendix describes procedures to be used when SW-846 Method 8000B is applied to the analysis of polychlorinated biphenyls (PCB) by GC/ECD. This Appendix is to be applied when SW-846 Method 8082 is requested, and is applicable to extracts derived from any matrix which are prepared according to the appropriate TestAmerica sample extraction SOP (CORP-OP-0001NC). The PCBs are determined and quantitated as Aroclor mixes.

1.2. Table C-1 lists compounds, which are routinely determined by this method, and gives the Reporting Limits (RL) for each matrix. RLs given are based on the low level standard and the sample preparation concentration factors. Matrix interferences may result in higher RLs than those listed.

1.2.1.1. Note: SW-846 method 8082 provides incomplete guidance for determination of individual PCB congeners. This SOP does not include directions for congener specific analysis.

1.3. The associated LIMS method code is QH (8082).

2. SUMMARY OF METHOD

2.1. This method presents conditions for the analysis of prepared extracts of PCBs. The PCBs are injected onto the column and separated and detected by electron capture detection. Quantitation is by the external standard method.

3. DEFINITIONS

3.1. Refer to the TestAmerica North Canton Laboratory Quality Manual (LQM), current version, for definitions of terms used in this document.

4. INTERFERENCES

4.1. Refer to the method 8000B section of this SOP for information regarding chromatographic interferences.

4.2. Interferences in the GC analysis arise from many compounds amenable to gas chromatography that give a measurable response on the electron capture detector. Phthalate esters, which are common plasticizers, can pose a major problem in the determinations. Avoiding contact with any plastic materials minimizes interferences from phthalates.

4.3. Sulfur will interfere and can be removed using procedures described in SOP NC-OP-0025.

4.4. Interferences co-extracted from samples will vary considerably from source to source. The presence of interferences may raise quantitation limits for individual samples. Specific cleanups may be performed on the sample extracts including, but not limited to Mercury, Tetrabutylammonium sulfite (TBA), Sulfuric Acid Cleanup, Silica Gel Cleanup, and Florisil® Cleanup of semivolatile organic compound extracts based on SW846 Methods 3660B, 3665A, 3630C, and 3620B. These cleanup procedures are included in SOP NC-OP-0025.

5. SAFETY

5.1. Refer to Section 5 of the Method 8000B SOP for general safety requirements.

5.2. Aroclors have been classified as a potential carcinogen under OSHA. Concentrated solutions of Aroclors must be handled with extreme care to avoid excess exposure. Contaminated gloves and

clothing must be removed immediately. Contaminated skin surfaces must be washed thoroughly.

- 5.3. All ^{63}Ni sources shall be leak tested every six months, or in accordance with the manufacturer's general radioactive material license.
- 5.4. All ^{63}Ni sources shall be inventoried every six months. If a detector is missing, the Director, EH&S shall be immediately notified and a letter sent to the NRC or local state agency.

6. EQUIPMENT AND SUPPLIES

- 6.1. Refer to Section 6 of the 8000B section of this SOP. A ^{63}Ni electron capture detector is required.
- 6.2. Refer to Table C-2 for analytical columns.
- 6.3. Microsyringes, various sizes, for standards preparation, sample injection, and extract dilution.

7. REAGENTS AND STANDARDS

- 7.1. Refer to the method 8000B section of this SOP for general requirements for reagents and supplies. The standards must be replaced at least every six months or sooner if comparison with check standards indicates a problem.
- 7.2. Refer to Table C-3 for details of calibration standards.
- 7.3. Surrogate Standards
 - 7.3.1. Tetrachloro-m-xylene and decachlorobiphenyl are the surrogate standards. Refer to Table C-4 for details of surrogate standards.

8. SAMPLE COLLECTION, PRESERVATION AND STORAGE

- 8.1 Refer to Section 8 of the 8000B section of this SOP.

9. QUALITY CONTROL

- 9.1 Refer to Section 9 of the 8000B section of this SOP.

10. CALIBRATION AND STANDARDIZATION

- 10.1. Refer to Section 10 of the 8000B section of this SOP for general calibration requirements.
- 10.2. Initial Calibration
 - 10.2.1. Refer to Table C-6 for the initial calibration analytical sequence.
 - 10.2.2. The response for each Aroclor will be calculated by the procedures described in the general method for GC analysis, with the following modifications.
 - 10.2.3. A minimum five-point calibration of all Aroclors is generated. The average response factor is used to quantitate Aroclors. The low level standard must be at or below the reporting limit. The other standards define the working range of the detector.
 - 10.2.4. The high and low standards for the initial five-point calibration of 1016 / 1260 define the acceptable quantitation range for the other Aroclors. If any Aroclor is determined above this concentration the extract must be diluted and reanalyzed.

- 10.2.4.1. NOTE: For Ohio VAP, Aroclor 1268 may be analyzed. In order to meet project specific reporting limits, a lower concentration standard may be added to the calibration curve.
- 10.2.5. If the analyst knows that a specific Aroclor is of interest for a particular project, that Aroclor may be used for the five point calibration rather than the 1016 / 1260 mix.
- 10.2.6. The surrogate calibration curve is calculated from the Aroclor 1016/1260 mix. Surrogates in the other calibration standards are used only as retention time markers.
- 10.2.7. The following is used for the quantitation of all Aroclors. The same quantitation option must be used for standards and samples.
- 10.2.7.1. Multiple peak option.
- 10.2.7.2. Select 3-10 major peaks in the analyte pattern. Calculate the response using the total area or total height of these peaks.
- 10.3. 12-Hour Calibration
- 10.3.1. The 12 -our calibration verification must be analyzed within 12 hours of the start of the initial calibration and at least once every 12 hours thereafter if samples are being analyzed. If there is a break in the analytical sequence of greater than 12 hours, then a new continuing calibration run must be analyzed before proceeding with the sequence. If more than 12 hours have elapsed since the injection of the last sample in the analytical sequence, a new analytical sequence must be started with a 12 hour calibration.
- 10.3.2. At a minimum, the 12-hour calibration includes analysis of the Aroclor 1260 / 1016 mix.
- 10.3.3. Other Aroclors are included in the daily calibration check.
- 10.3.4. The retention time windows for any analytes included in the daily calibration and CCVs are updated.
- 10.3.5. For this method samples must be bracketed with successful calibration verification runs.
- 10.4. Calibration verification standards
- 10.4.1. The Aroclor 1260/1016 calibration mix is analyzed as the calibration verification standard. This is analyzed after every 20 samples, including matrix spikes, LCS, and method blanks. (Depending on the type of samples, it may be advisable to analyze verifications more frequently in order to minimize reruns.).
- 10.4.2. A mid level standard is used for the calibration verification.

11. PROCEDURE

- 11.1. Refer to the Method 8000B section of this SOP for general procedural requirements.
- 11.2. Extraction
- 11.2.1. The extraction procedure is described in SOP CORP-OP-0001NC.
- 11.3. Cleanup
- 11.3.1. Cleanup procedures are described in SOP NC-OP-0025.

11.4. Suggested gas chromatographic conditions are given in Table C-2.

11.5. Allow extracts to warm to ambient temperature before injection.

11.6. The suggested analytical sequence is given in Table C-6.

12. DATA ANALYSIS AND CALCULATIONS

12.1. Identification of Aroclors

12.1.1. Retention time windows are used for identification of Aroclors, but the “fingerprint” produced by major peaks of those analytes in the standard is used in tandem with the retention times for identification. The ratios of the areas of the major peaks are also taken into consideration. Identification may be made even if the retention times of the peaks in the sample fall outside of the retention time windows of the standard, if in the analyst’s judgment the fingerprint (retention time and peak ratios) resembles the standard chromatogram.

12.1.2. A clearly identifiable Aroclor pattern serves as confirmation of single column GC analysis. Dual column confirmation may be used for specific program requirements or by client request.

12.2. Quantitation of Aroclors

12.2.1. Use 3-10 major peaks or total area for quantitation

12.2.2. If the analyst believes that a combination of Aroclor 1254 and 1260, or a combination of 1242, 1248 and 1232 is present, then only the predominant Aroclor is quantitated and reported, but the suspicion of multiple Aroclors is discussed in the narrative. If well separated Aroclor patterns are present, and then multiple Aroclors may be quantitated and reported.

12.3. Second column confirmation of Aroclors will only be performed when requested by the client. The appearance of the multiple peaks in the sample usually serves as a confirmation of Aroclor presence.

12.4. Surrogate recovery results are calculated and reported for decachlorobiphenyl (DCB) and tetrachloro-m-xylene (TCMX). Corrective action is only necessary if DCB and TCMX are both outside of acceptance limits, or if one is <10%.

12.4.1. NOTE: For Ohio VAP samples and DoD projects, all surrogates must meet acceptance limits.

13. METHOD PERFORMANCE

13.1. Performance limits for the four replicate initial demonstration of capability are required as referenced under Section 13.1 of the main body of this SOP.

13.2. Method detection limits (MDL) are determined for all Aroclors.

14. POLLUTION PREVENTION

14.1. Refer to Section 14 of the 8000B section of this SOP.

15. WASTE MANAGEMENT

15.1. Refer to Section 15 of the 8000 section of this SOP

15.2. Waste generated in this procedure will be segregated and disposed according to the facility hazardous waste procedures. The Environmental Health and Safety Director should be contacted if additional

information is required.

16. REFERENCES

16.1. SW846, Update III, December 1996, Method 8082

17. MISCELLANEOUS

17.1. Modifications from Reference Method

17.1.1. Method 8082 includes limited direction for congener specific quantitation. This is outside the scope of this SOP.

17.2. TABLES

Table C-1 Standard Analyte list and Reporting Limits for Method 8082			
Compound	Reporting Limit, µg/L or µg/kg		
	Water	Soil	Waste
Aroclor-1016	1.0	33	1000
Aroclor-1221	1.0	33	1000
Aroclor-1232	1.0	33	1000
Aroclor 1242	1.0	33	1000
Aroclor-1248	1.0	33	1000
Aroclor-1254	1.0	33	1000
Aroclor-1260	1.0	33	1000

The following concentration factors are assumed in calculating the Reporting Limits:

	<u>Extraction Vol.</u>	<u>Final Vol.</u>
Ground water	1000 mL	10 mL
Low-level Soil	30 g	10 mL
High-level soil / waste	1 g	10 mL

Table C-2 Instrumental Conditions for Method 8082	
Parameter	Recommended Conditions
Injection port temp	220°C
Detector temp	325°C
Temperature program	70°C for 0.5min, 30°C/min to 190°C, 2.5°C/min to 225, 18°C/min to 280°C, 3 min hold
Column 1	DB-5 or Rtx-5 30m x 0.32mm id, 0.5µm
Column 2	DB-1701 or Rtx 1701 30m x 0.32 mm id, 0.25µm
Column 3	DB-608, 30m X 0.32 mm, 0.25µm
Injection	1-2µL
Carrier gas	Helium or Hydrogen
Make up gas	Nitrogen
Y splitter	Restek or J&W or Supelco glass tee

Table C-3 Calibration Levels ng/mL for Method 8082						
	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6 ¹
Aroclor 1016/1260	0.05	0.1	0.2	0.5	1.0	2.0
Aroclor 1242 ²	0.05	0.1	0.2	0.5	1.0	2.0
Aroclor 1221 +1254 ²	0.05	0.1	0.2	0.5	1.0	2.0
Aroclor 1232 ²	0.05	0.1	0.2	0.5	1.0	2.0
Aroclor 1248 ²	0.05	0.1	0.2	0.5	1.0	2.0
Aroclor 1262	0.05	0.1	0.2	0.5	1.0	2.0
Aroclor 1268	0.05	0.1	0.2	0.5	1.0	2.0
Surrogates are included with all the calibration mixes at the following levels:						
Tetrachloro-m-xylene	5	10	25	50	100	200
Decachlorobiphenyl	5	10	25	50	100	200

¹ Level 6 is optional and should only be used if linearity can be maintained on the instrument to this level.

² Aroclors may be quantitated within the range 100 to 2000 ng/mL (4000ng/mL if the level 6 1016/1260 standard is included). If the Aroclor is more concentrated, it must be reanalyzed at a dilution.

Table C-4 LCS/Matrix Spike and Surrogate Spike levels for Aroclor analysis mg/L or mg/kg for Method 8082			
	Aqueous	Soil	Waste
Aroclor 1016/1260	10	333	10,000
Tetrachloro-m-xylene (Surrogate)	0.20	6.67	200
Decachlorobiphenyl (Surrogate)	0.20	6.67	200

Table C-5 Michigan Analyte List and Reporting Limits¹ for Method 8082			
Compound	Reporting Limit		
	water (µg/L)	soil (µg/Kg)	
Aroclor-1016	0.2	330	
Aroclor-1221	0.2	330	
Aroclor-1232	0.4	330	
Aroclor 1242	0.2	330	
Aroclor-1248	0.2	330	
Aroclor-1254	0.2	330	
Aroclor-1260	0.2	330	

¹ Reporting Limits are only for samples performed under the Michigan program

Table C-6
Suggested Analytical Sequence for Method 8082

Initial Calibration

Injection

1	Solvent blank (optional)	
2	Aroclor 1016/1260	Level 1
3	Aroclor 1016/1260	Level 2
4	Aroclor 1016/1260	Level 3
5	Aroclor 1016/1260	Level 4
6	Aroclor 1016/1260	Level 5
7	Aroclor 1232	An Initial Calibration includes Levels 1-5. A midpoint is used as CCV.
8	Aroclor 1242	An Initial Calibration includes Levels 1-5. A midpoint is used as CCV.
9	Aroclor 1248	An Initial Calibration includes Levels 1-5. A midpoint is used as CCV.
10	Aroclor 1221/1254	An Initial Calibration includes Levels 1-5. A midpoint is used as CCV.
11	Aroclor 1268 or 1262	An Initial Calibration includes Levels 1-5. A midpoint is used as CCV.
12	ICV	
13-32	Sample 1-20 (or as many samples as can be analyzed in 12 hours)	
33	Aroclor 1016/1260	Level 3

etc

Note: A solvent blank or primer may be analyzed at any time during the sequence when highly contaminated samples are expected. A solvent blank or primer may not be analyzed as routine immediately prior to standards.

12-hour Calibration

At least every 12 hours, counting from the start of the initial calibration, or from the start of the last daily calibration, the retention time windows must be updated using the Aroclor 1260 / 1016 mix. Mid level standards of any other Aroclors expected to be present in the samples are also injected.

1. SCOPE AND APPLICATION

- 1.1. This method is applicable to the gas chromatographic determination of Chlorinated phenoxy acid herbicides in extracts prepared by SOP NC-OP-0031NC. The herbicides listed in Table D1 are routinely analyzed. Other chlorinated acids may be analyzed by this method if the quality control criteria in Section 9 and the initial demonstration of method performance in Section 13 are met.
- 1.2. The associated LIMS method code is QS.

2. SUMMARY OF METHOD

- 2.1. This method presents conditions for the analysis of prepared extracts of phenoxy acid herbicides by gas chromatography. The herbicides, as their methyl esters, are injected onto the column, separated, and detected by electron capture detectors. Quantitation is by the external standard method.

3. DEFINITIONS

- 3.1 Refer to the TestAmerica North Canton Laboratory Quality Manual (LQM), current version, for definitions of terms used in this document.

4. INTERFERENCES

- 4.1. Refer to the method 8000B section of this SOP for general information regarding chromatographic interferences.
- 4.2. Chlorinated acids and phenols cause the most direct interference with this method.
- 4.3. Sulfur may interfere and may be removed by the procedure described in SOP# NC-OP-0025.

5. SAFETY

- 5.1. Refer to Section 5 of the Method 8000B SOP for general safety requirements.

6. EQUIPMENT AND SUPPLIES

- 6.1. Refer to Section 6 of the 8000B section of this SOP. A Ni₆₃ electron capture detector is required.
- 6.2. Refer to Table D2 for analytical columns.
- 6.3. Microsyringes, various sizes, for standards preparation, sample injection, and extract dilution.

7. REAGENTS AND STANDARDS

- 7.1. Refer to Section 7 of the 8000B section of this SOP for general information on reagents and standards.

8. SAMPLE PREPARATION, PRESERVATION AND STORAGE

- 8.1 Refer to Section 8 of the 8000B section of this SOP.

9. QUALITY CONTROL

- 9.1. Refer to Section 9 of the 8000B section of this SOP for quality control requirements, including the initial demonstration of capability, definition of a batch, surrogate limits, method blanks, laboratory control

samples (LCS), and matrix spikes (MS).

9.2. Refer to Table D-3 for the components and levels of the LCS and MS mixes.

10. CALIBRATION AND STANDARDIZATION

10.1. Refer to Section 10 of the 8000B section of this SOP for general calibration requirements.

10.2. Calibration standards are prepared from purchased standards in the methyl ester form.

10.3. The low level standard must be at or below the laboratory reporting limit. Other standards are chosen to bracket the expected range of concentrations found in samples, without saturating the detector or leading to excessive carryover.

10.4. Refer to Table D-2, for details of GC operating conditions.

11. PROCEDURE

11.1. Refer to the Method 8000B section of this SOP for procedural requirements.

11.2. Extraction

11.2.1. The extraction procedure is described in SOP #CORP-OP-0001NC.

11.3. Cleanup

11.3.1. The alkaline hydrolysis and subsequent extraction of the basic solution described in the extraction procedure provides an effective cleanup.

11.4. Analytical Sequence

11.4.1. The analytical sequence starts with an initial calibration of at least five points, or a daily calibration that meets % difference criteria from an existing initial calibration.

11.4.2. The daily calibration must be analyzed at least once every 24 hours when samples are being analyzed. If there is a break in the analytical sequence of greater than 12 hours, then a new continuing calibration run must be analyzed before proceeding with the sequence.

11.4.3. The daily calibration consists of mid level standards of all analytes of interest. Retention time windows must be updated with the daily calibration.

11.4.4. After every 12 hours a continuing calibration is analyzed. The continuing calibration consists of mid level standards of all analytes of interest. Retention time windows are updated with continuing calibrations.

11.5. Gas Chromatography

11.5.1. Chromatographic conditions are listed in Table D-2.

12. DATA ANALYSIS AND CALCULATIONS

12.1. Refer to the 8000B section of this SOP for identification and quantitation of single component analytes.

12.2. The herbicides are analyzed as their methyl esters, but reported as the free acid. For this reason it is necessary to correct the results for the molecular weight of the ester versus the free acid. This is achieved through the concentrations of the calibration standards. For example the 20µg/L calibration

standard for 2,4-D contains 21.3 µg/L of the methyl ester. No further correction is necessary.

- 12.3. A routine 10X dilution occurs on final extracts for all samples. Due to a QuantIMS limitation, the dilution factor field in QuantIMS cannot be used when a dilution is routine, because the dilution factor is automatically applied to all reference values creating reporting problems. For the herbicide analysis, the extract volume will be 10mL and an aliquot at 10X dilution will be analyzed. The final extract volume recorded on the laboratory bench sheet will be recorded as 100mL to avoid using the dilution factor field in QuantIMS.

13. METHOD PERFORMANCE

- 13.1. The EPA for this method has not published multiple laboratory performance data. Performance limits for the four replicate initial demonstration of capability are required as referenced under Section 13.1 of the main body of this SOP.

14. POLLUTION PREVENTION

- 14.1. This method does not contain any specific modifications that serve to minimize or prevent pollution.

15. WASTE MANAGEMENT

- 15.1. Waste generated in this procedure will be segregated and disposed according to the facility hazardous waste procedures. The Environmental Health and Safety Director should be contacted if additional information is required.

16. REFERENCES

- 16.1. Method 8151A, SW-846, Update III, December 1996

17. MISCELLANEOUS

- 17.1. Modifications from Reference Method
- 17.1.1. Refer to the method 8000B section of this SOP for modifications from the reference method.
- 17.2. Modifications from Previous Revision
- 17.2.1. The calibration procedure has been changed to require esterification of the calibration standards

17.3. TABLES

Table D-1				
Standard Analyte List for Method 8151A				
Compound	CAS Number	Reporting Limit, µg/L or µg/kg		
		Aqueous	Soil	Waste
2,4-D	94-75-7	4	80	4000
2,4-DB	94-82-6	4	80	4000
2,4,5-TP (Silvex)	93-72-1	1	20	1000
2,4,5-T	93-76-5	1	20	1000
Dalapon	75-99-0	2	40	2000
Dicamba	1918-00-9	2	40	2000
Dichloroprop	120-36-5	4	80	4000
Dinoseb	88-85-7	0.6	12	600
MCPA	94-74-6	400	8000	400,000
MCPD	93-65-2	400	8000	400,000

The following concentration factors are assumed in calculating the Reporting Limits:

	<u>Extraction Vol.</u>	<u>Final Vol.</u>	<u>Dilution Factor</u>
Ground water	1000 mL	10 mL	10
Low-level Soil without GPC	50 g	10 mL	10
High-level soil / waste	1 g	10 mL	10

Specific reporting limits are highly matrix dependent. The reporting limits listed above are provided for guidance only and may not always be achievable. For special projects, the extracts may be analyzed without any dilution, resulting in reporting limits 20 times lower than those in Table D-1.

Table D-2	
Instrumental Conditions for Method 8151A	
PARAMETER	Recommended conditions
Injection port temp	220°C
Detector temp	325°C
Temperature program	80,2/30/170,0/1/180,1
Column 1	DB-5MS or RTX 5 30x0.32, 0.5µm
Column 2	DB-1701 or Rtx-1701
Injection	1-2µL
Carrier gas	Helium / Hydrogen
Make up gas	Nitrogen

Recommended conditions should result in resolution of all analytes listed in Table D-1.

The reporting limits listed in Table D-1 will be achieved with these calibration levels and a 20-fold dilution of the sample extract. Lower reporting limits can be achieved with lesser dilutions of the sample extract.

Table D-3			
LCS/Matrix Spike and Surrogate Spike levels mg/L or mg/kg¹ for Method 8151A			
	Aqueous	Soil	Waste
2,4-D	40	400	20000
Silvex	10	100	5000
2,4,5-T	10	100	5000
2,4-DB	40	400	20000
Dalapon	20	200	10000
DCAA (surrogate)	40	400	20000
Dicamba	20	200	10000
MCPP	4000	40000	200000
MCPA	4000	40000	200000
Dichloroprop	40	400	2000
Pentachlorophenol	5	50	2500
Dinoseb	6	60	300

¹ LCS, MS and SS spikes are as the free acid.

1. SCOPE AND APPLICATION

- 1.1. This method is applicable to the determination of the concentration and **tentative** identification of extractable petroleum (diesel range) hydrocarbon mixes in waters, wastewaters, soils, and sludges.
- 1.2. This SOP is based on SW-846 Method 8015B, Modified, Revision 3, December 1996.
- 1.3. The associated LIMS method codes are HS (8015 MOD) and KI (8015B).

2. SUMMARY OF METHOD

- 2.1. This method provides gas chromatographic conditions for detection and identification of total petroleum hydrocarbons. Prior to the use of this method, appropriate sample preparation techniques are used.
- 2.2. An aliquot of the prepared sample is injected into a gas chromatograph (GC) and compounds in the effluent are detected by a flame ionization detector (FID).
- 2.3. The laboratory carbon range for Ohio VAP projects is C10-C20 and C20-C32. The laboratory carbon range for BUSTR Projects is C10-C20 and C20-C34.

3. DEFINITIONS

- 3.1. Refer to the glossary in the TESTAMERICA North Canton Laboratory Quality Manual (LQM), current version.

4. INTERFERENCES

- 4.1. Refer to the method 8000B section of this SOP for general information regarding chromatographic interferences.

5. SAFETY

- 5.1. Refer to Section 5 of the Method 8000B SOP for general safety requirements.

6. EQUIPMENT AND SUPPLIES

- 6.1. Refer to Section 6 of the 8000B section of this SOP.
- 6.2. Microsyringes, various sizes, for standards preparation, sample injection, and extract dilution.

7. REAGENTS AND STANDARDS

- 7.1. Refer to Section 7 of the 8000B section of this SOP.
- 7.2. The petroleum hydrocarbons are purchased from a chemical supplier when available. When no chemical supplier is available, the fuels are purchased from public sources.
- 7.3. The OVAP and BUSTR standard is a commercially prepared standard containing alkanes from C10-C34.

8. SAMPLE PREPARATION, PRESERVATION AND STORAGE

- 8.1. Refer to Section 8 of the 8000B section of this SOP.

9. QUALITY CONTROL

- 9.1. Refer to Section 9 of the 8000B section of this SOP for quality control requirements, including the initial demonstration of capability, definition of a batch, surrogate limits, method blanks, laboratory control samples (LCS), and matrix spikes (MS).
- 9.2. MS/MSD recoveries are calculated from a Diesel calibration.
- 9.3. Surrogates
- 9.3.1. Because of the nature of the TPH analysis, whereas certain petroleum mixtures can override the C9 (Nonane) surrogate. The C9 (Nonane) surrogate recoveries are advisory. Re-extraction due to surrogate recoveries is determined by analyst judgement.
- 9.3.1.1. NOTE: Ohio VAP rules require reanalysis when surrogate recoveries are outside of control limits.

10. CALIBRATION AND STANDARDIZATION

Refer to Section 10 of the 8000B section of this SOP for general calibration requirements.

- 10.1. Recommended Instrument Conditions
- 10.1.1. Hydrogen carrier gas - flow rate 5 - 6 mL/min
- 10.1.2. Detector gas mixture - air hydrogen mixture in a 10:1 ratio, air 80 - 120 mL/min, hydrogen 8 - 12 mL/min
- 10.1.3. Temperature Program - refer to Table F1 Appendix
- 10.1.4. Injection volume - 1 µL
- 10.2. Initial Calibration
- 10.2.1. Analyze a five point Diesel calibration standard referring to the recommended instrument conditions. The calibration concentrations are 100, 200, 500, 1000, and 2000 ng/uL. A 5000ng/uL standard may be analyzed if needed. The retention time window of C10-C32 shall be used for the Diesel calibration. The low level standard must be at or below the reporting limit. The other standards define the working range of the detector.
- 10.2.2. For Ohio VAP and BUSTR projects, the laboratory analyzes a five point calibration for the carbon range C10-C20. The concentrations are 60, 120, 240, 600 and 1200 ug/mL. In addition, a five point of the carbon range C20-C34 is also analyzed. The concentration ranges are 80, 160, 320, 800, and 1600 ug/mL.
- 10.3. Continuing Calibration
- 10.3.1. Refer to Section 10 of the 8000B section of this SOP for general calibration requirements.
- 10.3.2. A mid-range standard of Diesel, C10-20, and C20-34 is used, as appropriate, for the CCV. The acceptance criteria is 15%.

11. PROCEDURE

- 11.1. Refer to the method 8000B section of this SOP for procedural requirements.
- 11.2. Extraction
 - 11.2.1. The extraction procedure is described in SOP #CORP-OP-0001NC.
- 11.3. Analytical Sequence – Refer to Section 11 in the 8000B Section of this SOP.
- 11.4. Petroleum Hydrocarbon Identification and/or Fingerprinting
 - 11.4.1. To identify the type of petroleum hydrocarbon, compare the chromatographic peak pattern to the patterns of known petroleum hydrocarbons analyzed under identical chromatographic conditions. Samples are quantified against diesel, but fingerprinting may be done when client requested.
 - 11.4.2. Positive matching may not be possible, even using site-specific hydrocarbons. Degradation of the pattern can occur during environmental exposure of the fuel. See Table 2 for possible fingerprints.
 - 11.4.3. Sample Quantification
 - 11.4.4. Samples are quantified against the initial calibration of diesel or DRO on a single column.
 - 11.4.5. The total height or area of the hydrocarbon is determined in the same manner used for the hydrocarbon standard.
 - 11.4.6. If the amount of sample injected into the GC exceeds the working range of the calibration curve, an appropriate dilution is performed before reanalysis.

12. DATA ANALYSIS AND CALCULATIONS

- 12.1. Refer to the 8000B section of this SOP for identification and quantitation of single component analytes.
- 12.2. Surrogate recovery results are calculated and reported for Nonane (C-9). The surrogate must be within QC criteria. Corrective action is only necessary if Nonane (C-9) is outside of acceptance limits.

13. METHOD PERFORMANCE

- 13.1. Performance limits for the four replicate initial demonstration of capability are required as referenced under Section 13.1 of the main body of this SOP.

14. POLLUTION PREVENTION

- 14.1. Refer to Section 14 of the 8000B section of this SOP.

15. WASTE MANAGEMENT

- 15.1. Waste generated in this procedure will be segregated and disposed according to the facility hazardous waste procedures. The Environmental Health and Safety Director should be contacted if additional information is required.

16. REFERENCES

- 16.1. SW846, Method 8015B, Nonhalogenated Organics Using GC/FID, Test Methods for Evaluating Solid Waste, Third Edition, USEPA
- 16.2. Related SOP
- 16.2.1. CORP-OP-0001NC, Extraction of Organic Compounds from Waters and Soils, Based on SW846 3500 Series, 3600 Series, 8150, 8151, and 600 Series Methods

Table E-1
Suggested GC Temperature Program for TPH Analysis

Initial Temperature	40°C
Initial Hold Time	4 minutes
Temperature Program	10°C/minute
Final Temperature	280°C
Final Hold Time	10 minutes

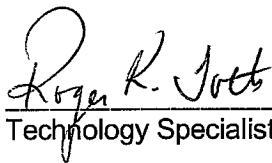
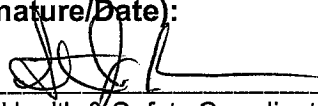
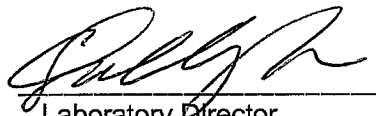
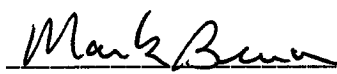
Table E-2
Reporting Limits for TPH Analysis

Analyte	Reporting Limits		
	Water (mg/L)	Solids (mg/kg)	Waste Dilution (mg/kg)
TPH (as Diesel) or DRO	100	3.3	200
C10-C20 (OVAP & BUSTR)	60	2.0	
C20-C34 (OVAP & BUSTR)	80	2.3	
Fingerprint Compounds ¹			
Mineral Spirits	Kerosene	Motor Oil	
Hydraulic Oil	Jet Fuel	Stoddard Solvent	
DRO Spiking Solution			
Decane	Dodecane	Tetradecane	
Hexadecane	Octobecane	Eicosane	
Docosane	Tetracosane	Hexacosane	
Octacosane			

¹ This list represents most of the common petroleum hydrocarbons. The list may be expanded to include other petroleum hydrocarbons.

Title: ACID DIGESTION FOR SOIL SAMPLES

[SW846 Method 3050B]

Approvals (Signature/Date):			
	7-2-08		7-03-08
Technology Specialist	Date	Health & Safety Coordinator	Date
	7/8/08		7/7/08
Quality Assurance Manager	Date	Laboratory Director	Date
	7/7/08		
Technical Director	Date		

This SOP was previously identified as SOP No. CORP-IP-0002NC, Rev 2.6, dated 07/29/07

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TABLE OF CONTENTS

1. SCOPE AND APPLICATION.....3

2. SUMMARY OF METHOD 3

3. DEFINITIONS.....3

4. INTERFERENCES.....3

5. SAFETY.....4

6. EQUIPMENT AND SUPPLIES 6

7. REAGENTS AND STANDARDS 7

8. SAMPLE COLLECTION, PRESERVATION AND STORAGE.....7

9. QUALITY CONTROL.....8

10. CALIBRATION AND STANDARDIZATION 10

11. PROCEDURE.....10

12. DATA ANALYSIS AND CALCULATIONS 13

13. METHOD PERFORMANCE.....13

14. POLLUTION PREVENTION 13

15. WASTE MANAGEMENT 13

16. REFERENCES.....14

17. MISCELLANEOUS (TABLES, APPENDICES, ETC.) 14

LIST OF APPENDICES:

APPENDIX A - TABLES 15

APPENDIX B - CONTAMINATION CONTROL GUIDELINES 21

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 (ICP/MS) as specified in SW846 Method 3050B.
- 1.2. Samples prepared by the protocols detailed in this SOP may be analyzed by ICP or ICP/MS 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 and sediments.
- 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 Perkin Elmer 6100 ICP/MS (Ag, Sb, Sn) 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.
- 3.2. Total Metals: The concentration determined on an unfiltered sample following digestion. Note that this method is designed to determine the total *environmentally available* metals.

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.

- 4.2. The entire work area, including the bench top and fume hood, should be thoroughly cleaned on a routine schedule in order to minimize the potential for environmental contamination.
- 4.3. Boron and silica from the glassware will grow into the sample solution during and following sample processing. For critical low level determinations of boron and silica, only quartz and/or plastic labware should 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. If physical interferences are present, they should be documented.
- 4.5. Visual interferences or anomalies, such as foaming, emulsions, precipitates, etc., 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 media.
- 4.7. 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 Safety 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 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
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.
Nitric Acid	Corrosive Oxidizer Poison	2 ppm-TWA 4 ppm-STEEL	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 will be removed and discarded; other gloves will 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 should 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**. Therefore, unless they are known to be non-hazardous, all samples should be opened, transferred and prepared in a fume hood, or under other means of mechanical ventilation. 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 will be kept closed unless transfers are being made.
- 5.7 The preparation of standards and reagents will be conducted in a fume hood with the sash closed as far as the operation will permit or under other means of mechanical ventilation.

- 5.8 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.
- 5.9 Always carry bulk concentrated acid bottles in appropriate impact proof containers.
- 5.10 Acid/peroxide spills must be neutralized immediately, flushed with water and cleaned up using appropriate spill kits.
- 5.11 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, 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. Griffin beakers of assorted sizes or equivalent
- 6.4. Vapor recovery device (Watch glasses, ribbed or other device)
- 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 100 mL within 3% accuracy
- 6.8. Analytical balance capable of accurately weighing to the nearest 0.01 grams
- 6.9. Repipetters or suitable reagent dispensers
- 6.10. Calibrated automatic pipettes with corresponding pipette tips or Class A glass volumetric pipettes, 100uL, 500uL, 1mL-5mL
- 6.11. Class A volumetric flasks
- 6.12. Plastic bottles, 120-150mL or equivalent
- 6.13. 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.
- 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% HNO₃. This acid (5 mL of concentrated HNO₃ 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. ICPMS LCS/MS spike solution. Laboratory Control Sample (LCS) and matrix spike (MS) solutions are custom made with the exception of Ag, Sb, and Sn (for the Perkin Elmer Elan 6100 ICPMS analysis), so that the final concentrations after spiking equals the concentrations listed in Tables III and IV. The ICP working solution (Section 7.3) is used for the Ag, Sb, and Sn spiking solution for the Perkin Elmer Elan 6100 Analysis, which is also 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 (HNO₃), concentrated, trace metal grade or better
- 7.7. Nitric acid, 1:1 - dilute concentrated HNO₃ 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 (H₂O₂), reagent 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 collection to the date of analysis.
- 8.2. Soil samples do not require preservation with the exception of mercury, which must be stored at $4\text{ }^{\circ}\text{C} \pm 2\text{ }^{\circ}\text{C}$ until time of preparation.

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 MDL is determined using seven replicates of reagent water, spiked with all the analytes of interest, that have been carried through the entire analytical procedure. MDLs must be redetermined on an annual basis in accordance with 40 CFR Part 136 Appendix B requirements or verified as detailed in TestAmerica Corporate CA-Q-S-006 and TestAmerica North Canton SOP NC-QA-021. The spike level must be between the calculated MDL and 10X the MDL to be valid. 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-0016.
- 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 should 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, a 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 SOP's. 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 1 g aliquot of boiling chips. The MB is then processed as described in Section 11.10.
- 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. On going 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 SOP's. Corrective action when LCS results fail to meet control limits will be reparation and reanalysis of the batch. Tables II and III provide the details regarding the stock, working standards and final spike concentrations for ICP and ICP/MS. Refer to Section 7.4 for instructions on preparation of the aqueous LCS.
- 9.5.1. The LCS is prepared by spiking a 1g aliquot of boiling chips with 2 mL of the working LCS/MS spike solution (Section 7.3) for ICP and the Perkin Elmer Elan 6100 ICPMS (Ag, Sb, Sn) analysis and 1 mL of the LCS/MS solution (Section 7.4) for the Thermo X Series 2 ICPMS analysis and the Perkin Elmer Elan 6100 analysis not requiring Ag, Sb, and Sn. The LCS is then processed as described in Section 11.10.
- 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 (DQO's) may require the use of sample duplicates in place of or in addition to MS/MSD's. 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 reparation and reanalysis of the batch. Corrective action when MS results fail to meet control limits does not include reparation of samples unless the results indicate that a spiking error may have occurred. Tables II through III provide

the details regarding the stock, working standards and final matrix spike concentrations for ICP and ICP/MS. Refer to Section 7.4 for instructions on preparation of the working matrix spike solutions.

9.6.1. The soil matrix spike sample is prepared by spiking a 1 g aliquot of a sample with 2mL of the working LCS/MS spike solution (Section 7.3) for ICP analysis and the Perkin Elmer Elan 6100 ICPMS (Ag, Sb, and Sn) analysis and 1 mL of the LCS/MS solution (Section 7.4) for the Thermo X Series 2 ICPMS analysis and the Perkin Elmer Elan 6100 analysis not requiring Ag, Sb, and Sn. The matrix spike sample is then processed as described in Section 11.10.

10. CALIBRATION AND STANDARDIZATION

10.1. Hotplate or hotblock temperature must be verified daily for each unit used, and must be recorded in a hotplate/hotblock temperature log. The hotplate temperature should be verified by measuring the temperature of a beaker of reagent water placed on each hotplate/hotblock. For block digestors, use a tube containing water.

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 shall be completely documented using a Nonconformance Memo. The Nonconformance Memo shall 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.
- 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 programs 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. 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 administrator for further instructions. In some cases, it may be more appropriate to process these samples as solids.

- 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. In some cases, both ICP/MS and ICP digestates are required on each sample. It is recommended that both aliquots be weighed out and processed at the same time.
- 11.9. Guidelines are provided in the appendices on procedures to minimize contamination of samples and standards.
- 11.10. Preparation of Soils, Sediments and Sludges for Analysis by ICP and ICP/MS
 - 11.10.1. Mix sample thoroughly by stirring with a clean plastic or wooden spoon or spatula.
 - 11.10.2. For each digestion procedure required (i.e., ICP or ICP/MS), 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.
 - 11.10.3. Measure additional aliquots of the designated samples for the MS and MSD analyses.
 - 11.10.4. Spike each of the MS and MSD aliquots with 2 mL of the working LCS/MS spiking solution (Section 7.3) for ICP analysis and the Perkin Elmer Elan 6100 ICPMS (Ag, Sb and Sn) analysis and 1 mL of the LCS/MS spiking solution (Section 7.4) for Thermo X Series 2 ICPMS analysis and the Perkin Elmer Elan 6100 analysis not requiring Ag, Sb and Sn.
 - 11.10.5. Prepare a beaker or equivalent container for the method blank.
 - 11.10.6. Prepare a beaker or equivalent container for the LCS. Add 2 mL of the working LCS/MS spiking solution (Section 7.3) for ICP analysis and the Perkin Elmer Elan 6100 (Ag, Sb and Sn) analysis and 1 mL of the LCS/MS spiking solution (Section 7.4) for ICP/MS analysis.
 - 11.10.7. Add 10 mL of 1:1 HNO₃ and mix the sample.
 - 11.10.8. Heat sample to 95° ±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. Allow sample to cool, if necessary.
 - 11.10.9. Add 5 mL of concentrated HNO₃.

- 11.10.10. Reflux at $95^{\circ} \pm 4^{\circ}$ C for 30 minutes. (Add reagent water as needed to ensure that the volume of solution is not reduced to less than 5 mL.)
- 11.10.11. Allow the samples to cool, if necessary.
- 11.10.12. 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.
- 11.10.13. Heat sample until effervescence subsides.
- 11.10.14. Allow the sample to cool, if necessary.
- 11.10.15. Continue adding 30% H_2O_2 in 1 mL aliquots with warming until effervescence is minimal or sample appearance is unchanged.

Note: Do not add more than a total of 10 mL of 30 % H_2O_2 .

- 11.10.16. Continue heating at $95^{\circ} \pm 4^{\circ}$ C until the volume is reduced to approximately 5-10 mL. Alternatively the sample may be heated for two hours for DoD projects.
- 11.10.17. If the sample is being prepared for ICP, Thermo X Series 2 ICPMS analysis or the Perkin Elmer Elan 6100 ICP/MS (Ag, Sb, Sn) analysis, add 10 mL of concentrated HCL and reflux for an additional 10 minutes (15 minutes for DoD projects) without boiling. This step is omitted for Perkin Elmer Elan 6100 ICP/MS elements not requesting Ag, Sb, and/or Sn.
- 11.10.18. Allow the sample to cool.
- 11.10.19. Wash down beaker walls and vapor recovery device with reagent water.
- 11.10.20. Filter sample through Whatman 41 filter paper or equivalent into a graduated cylinder or equivalent or a pre-weighed bottle. Other measuring bottles (for example, Corning Snap Seals™) may be used if their accuracy is documented and is better than $\pm 2\%$. Rinse beaker and filter paper with reagent water to ensure complete sample transfer.

Note: In place of filtering, the samples, after dilution and mixing, may be centrifuged or allowed to settle by gravity overnight to remove insoluble material

- 11.10.21. Dilute sample to 100 mL with reagent water. The sample is now ready for analysis.

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. This method does not contain any specific modifications that serve to minimize or prevent pollution.

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 Safety Manual 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 Safety Manual, M-E-0001, and TestAmerica North Canton Facility Addendum and Contingency Plan, current version

16.1.4. Revision History

<u>Historical File:</u>	Revision 2.1: 02/11/00	Revision 2.5: 12/02/04
(formerly known as	Revision 2.2: 09/25/01	Revision 2.6: 07/29/07
CORP-IP-0002NC)	Revision 2.3: 01/18/02	
	Revision 2.4: 02/19/03	

16.2. Associated SOPs and Policies, latest 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, CA-Q-S-006 and NC-QA-021

16.2.7. Supplemental Practices for DOD Project Work, NC-QA-0016

16.2.8. Standards and Reagents, NC-QA-017

17. **MISCELLANEOUS (TABLES, APPENDICES, ETC.)**

17.1. Modifications/Interpretations from reference method.

- 17.1.1. Chapter 1 of SW-846 states that the method blank should 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, as defined in the determinative SOPs, are allowed up to two times the reporting limit in the blank.

17.2. Documentation and Record Management

The preparation benchsheet should, at a minimum, include the following information:

- Preparation date, analyst, matrix, prep type (ICP or ICP/MS)
- Sample ID; initial weight/volume and final weight/volume
- Standards Documentation (source, lot, prep date, volume added)
- Reagents

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

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

Perkin Elmer Elan 6100 ICP/MS Soil Matrix Spike and LCS Levels

Element	Working LCS/MS Standard (mg/kg)	Soil MS/LCS Level * (mg/kg)
Antimony **	25	50
Arsenic	10	10
Barium	10	10
Beryllium	10	10
Cadmium	10	10
Chromium	10	10
Cobalt	10	10
Copper	10	10
Lead	10	10
Manganese	10	10
Molybdenum	10	10
Nickel	10	10
Selenium	10	10
Silver **	2.5	5
Strontium	10	10
Thallium	10	10
Vanadium	10	10
Zinc	10	10
Boron	10	10
Tin **	100	200
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).

** In order to reduce the amount of sample digestions, Ag, Sb, and Sn will be spiked at the same concentration as the ICP spike levels. A 10x dilution is recommended for the Perkin Elmer ICPMS analysis of the MS/MSD and LCS before analysis.

TABLE IV

Thermo X Series 2 ICPMS Soil Matrix Spike and LCS Levels

Element	Working LCS/MS Standard (mg/kg)	Soil MS/LCS Level * (mg/kg)
Aluminum	100	100
Antimony	10	10
Arsenic	10	10
Barium	10	10
Beryllium	10	10
Cadmium	10	10
Calcium	100	100
Chromium	10	10
Cobalt	10	10
Copper	10	10
Iron	100	100
Lead	10	10
Magnesium	100	100
Manganese	10	10
Molybdenum	10	10
Nickel	10	10
Potassium	100	100
Selenium	10	10
Silver	10	10
Sodium	100	100
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).

TABLE V

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 reanalyze 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 reanalyze 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

APPENDIX B

CONTAMINATION CONTROL GUIDELINES

APPENDIX B. CONTAMINATION CONTROL GUIDELINES

The following procedures are strongly recommended to prevent contamination:

All work areas used to prepare standards and spikes should be cleaned before and after each use.

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 must be kept scrupulously clean.

Powdered or latex gloves must not be used in the metals laboratory since the powder contains silica and zinc, as well as other metallic analytes. Only vinyl or nitrile gloves should be used in the metals laboratory.

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:

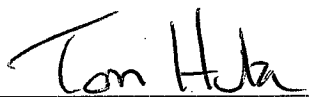
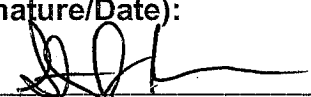
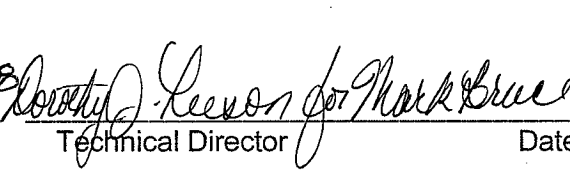
Reagents or standards can contain contaminants or be contaminated with the improper use of a pipette.

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.

Title: GC/MS ANALYSIS BASED ON METHOD 8270C

[Method 8270C]

Approvals (Signature/Date):	
 Technology Specialist	 Health & Safety Coordinator
5/14/08 Date	5/15/08 Date
 Quality Assurance Manager	 Technical Director
5/16/08 Date	5/16/08 Date
 Laboratory Director	
5/16/08 Date	

This SOP was previously identified as SOP CORP-MS-0001NC, Rev 2.12 dated 03/01/07

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TABLE OF CONTENTS

1. SCOPE AND APPLICATION	4
2 SUMMARY OF METHOD.....	4
3 DEFINITIONS.....	5
4 INTERFERENCES	5
5 SAFETY PRECAUTIONS	6
6 EQUIPMENT AND SUPPLIES	7
7 REAGENTS AND STANDARDS.....	8
8 SAMPLE PRESERVATION AND STORAGE.....	8
9 QUALITY CONTROL	8
10 CALIBRATION AND STANDARDIZATION	12
11 PROCEDURE	16
12 DATA ANALYSIS AND CALCULATIONS	18
13 METHOD PERFORMANCE	24
14 POLLUTION PREVENTION.....	24
15 WASTE MANAGEMENT.....	25
16 REFERENCES	25
17 MISCELLANEOUS	26

LIST OF TABLES

TABLE 1	TestAmerica North Canton Primary Standard and Standard Reporting Limits
TABLE 2	TestAmerica North Canton Appendix IX Standard and Standard Reporting Limits
TABLE 2A	TestAmerica North Canton Michigan Program
TABLE 3	Reportable Analytes for TestAmerica North Canton Standard Tests, Primary Standard
TABLE 4	Reportable Analytes for TestAmerica North Canton Standard Tests, Appendix IX Standard
TABLE 5	Suggested Instrument Conditions
TABLE 6	DFTPP Ion Abundance Criteria
TABLE 7	Characteristic Ions, Primary Standard
TABLE 8	Characteristic Ions, Appendix IX Standard
TABLE 9	8270C LCS Control Compounds
TABLE 9A	8270 All-Analyte Spike Mix
TABLE 10	TCLP LCS Compounds
TABLE 11	8270C Surrogate Compounds
TABLE 12	Calibration Ranges
TABLE 13	Calibration Ranges, Appendix IX
TABLE 14A, B	Semivolatile Internal Standards with Corresponding Analytes Assigned for Quantitation

1. SCOPE AND APPLICATION

- 1.1 This method is based upon SW846 8270C, 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, 2, 3, and 4 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 SRLs. Reporting limits will be proportionately higher for sample extracts that require dilution.
- 1.4 The associated LIMS code is QL (8270C).

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, soxhlet, accelerated soxhlet or pressurized fluid extraction. 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. Qualitative identification of the parameters in the extract is performed using the retention time and the relative abundance of characteristic ions. Quantitative analysis is performed using the internal standard technique with a single characteristic ion.

3. DEFINITIONS

- 3.1 CCC (Calibration Check Compounds) - A subset of target compounds used to evaluate the calibration stability of the GC/MS system. A maximum percent deviation of the CCCs is specified for calibration acceptance.
- 3.2 SPCC (System Performance Check Compounds) - Target compounds designated to monitor chromatographic performance, sensitivity, and compound instability or degradation on active sites. Minimum response factors are specified for acceptable performance.
- 3.3 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.
- 3.4 Method Blank - An analytical control consisting of all reagents, internal standards, and surrogate standards that is carried through the entire analytical procedure. The method blank is used to define the level of laboratory background and reagent contamination.
- 3.5 LCS (Laboratory Control Sample) - A blank spiked with the parameters of interest that is carried through the entire analytical procedure. Analysis of this sample with acceptable recoveries of the spiked materials demonstrates that the laboratory techniques for this method are acceptable.
- 3.6 MS (Matrix Spike)- Aliquot of a matrix (water or soil) fortified (spiked) with known quantities of specific compounds and subjected to the entire analytical procedure in order to indicate the appropriateness of the method for the matrix by measuring recovery.
- 3.7 MSD (Matrix Spike Duplicate)- A second aliquot of the same sample as the matrix spike (above) that is spiked in order to determine the precision of the method for the matrix by measuring relative percent difference.

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 should 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 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 become contaminated will be removed and discarded; other gloves will 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 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
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 degrades 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 Exposure to chemicals must be maintained as low as reasonably achievable; therefore, unless they are known to be non-hazardous, all samples must be opened, transferred, and prepared in a

fume hood, or under other means of mechanical ventilation. Solvent and waste containers must be kept closed unless transfers are being made.

- 5.6 The preparation of standards and reagents will be conducted in a fume hood with the sash closed as far as the operation will permit.
- 5.7 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.
- 5.8 Standards in solution may be diluted in the open laboratory when syringes and the like are utilized.
- 5.9 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 should be directly coupled to the source.
- 6.2 Column: 20m x 0.18mm ID, 0.36 μ m 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 6 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 μ L Hamilton Laboratory grade syringes or equivalent.
- 6.7 Carrier gas: Ultra high purity helium.

7. REAGENTS AND STANDARDS

- 7.1 A minimum five-point calibration curve is prepared. If a quadratic regression is used, six points must be analyzed for the calibration curve. The low point should be at or below the reporting limit. Refer to Tables 12 and 13 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. 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. See appropriate preparation SOP. Surrogate compounds and levels are listed in Table 11.
- 7.4 GC/MS Tuning Standard: A methylene chloride solution containing decafluorotriphenylphosphine (DFTPP) is prepared. Pentachlorophenol, benzidine, and DDT, should 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 $\leq 6^{\circ}\text{C}$ when not in use. Refrigeration at -10°C to -20°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.

8. SAMPLE PRESERVATION AND STORAGE

- 8.1 Sample extracts are stored at $4 \pm 2^{\circ}\text{C}$. Samples and extracts must be stored in suitable glass containers with Teflon lined caps. (Extracts will be stored for 30 days after invoicing.)
- 8.3 Water samples are extracted within seven days of sampling and the extracts are analyzed within forty days of extraction. Solids, sludges, and organic liquids are extracted within fourteen days of sampling and the extracts are analyzed within forty 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 should 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 shall include an MDL study analysis of your 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. The recovery limits are mean recovery ± 3 standard deviations for surrogates, MS and LCS. Precision limits for matrix spikes / matrix spike duplicates are mean relative percent difference ± 3 standard deviations. Control limits are established by the laboratory as described in SOP NC-QA-0018. Control limits are easily accessible via LIMs (QC Browser program).

9.2.2 If samples are diluted, the surrogate and matrix spike recoveries will be reported with a DIL flag. Any analyte outside of the control limits will be flagged with JDIL. For DoD projects all surrogates must be within control limits.

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 Method Blank

9.3.1 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.

9.3.1.1 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.

9.3.1.2 Reanalysis of any samples with reportable concentrations of analytes found in the method blank is required unless other actions are agreed with the client.

9.3.1.3 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 will be re-extracted and re-analyzed.

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 should take place. For Ohio VAP samples, all analytes must meet criteria or the samples must be re-extracted if sufficient volume of sample remains.

9.3.3 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 North Canton QC Program document (QA-003) for further details of the corrective actions.

9.4 Laboratory Control Sample (LCS)

9.4.1 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 Tables 9 and 10 unless specified by a client or agency.

9.4.2 If any control analyte in the LCS is outside the laboratory established historical control limits, corrective action must occur. All noncontrolling compounds must attain a recovery of 5% or greater if the compound is on the client's list. Corrective action may include re-extraction and reanalysis 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.

9.4.2.1 If the batch is not re-extracted and reanalyzed, the reasons for accepting the batch must be clearly presented in the project records and the report. (An example of acceptable reasons for not reanalyzing 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).

9.4.2.2 If re-extraction and reanalysis 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.

9.4.2.3 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 if sufficient volume of sample remains.

9.4.3 Ongoing monitoring of the LCS over time provides evidence that the laboratory is performing the method within accepted QC guidelines for accuracy and precision.

9.5 Matrix Spike/Matrix Spike Duplicate (MS/MSD)

9.5.1 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 Tables 9 and 10). Compare the percent recovery and relative percent difference (RPD) to that in the laboratory specific historically generated limits.

9.5.1.1 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 will normally include reparation and reanalysis of the batch.

9.5.1.2 If a MS/MSD is not possible due to limited sample, then a LCS duplicate should be analyzed. RPD of the LCS and LCSD are compared to the matrix spike limits.

9.5.1.3 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.

9.6 Surrogates

9.6.1 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 11.

9.6.2 If any surrogates are outside limits the following corrective actions must take place (except for dilutions):

9.6.2.1 Check all calculations for error.

9.6.2.2 Ensure that instrument performance is acceptable.

9.6.2.3 Recalculate the data and/or reanalyze the extract if either of the above checks reveal a problem.

9.6.2.3.1 It is only necessary to reprepare / reanalyze 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.

9.6.3 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 reanalysis as this phenomenon would indicate a possible matrix problem.

9.6.4 If the sample is reanalyzed and the surrogate recoveries in the reanalysis are acceptable, then the problem was within the analyst's control and only the reanalyzed data should be reported. (Unless the reanalysis was outside holding times, in which case reporting both sets of results may be appropriate.)

9.6.5 If the reanalysis does confirm the original results, the original analysis is reported and the data flagged as estimated due to matrix effect.

9.7 Nonconformance and Corrective Action

9.7.1 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 5.

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 twelve hour shift when analyses are to be performed, the GC/MS system must be checked to see if acceptable performance criteria (Table 6) 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 6 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. 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 7. 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.
- 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 Tables 12 and 13. 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. Calculate response factors (RF), average response factors, and the percent RSD of the response factors for each compound using the equations in Section 12 and verify that the CCC and SPCC criteria in Sections 10.4.5 and 10.4.6 are met. **No sample analysis may be performed unless these criteria are met.**
- 10.4.5 System Performance Check Compounds (SPCCs): 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

- 10.4.6 Calibration Check Compounds (CCCs): 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.

- 10.4.6.1 If none of the CCCs are required analytes, project specific calibration specifications must be agreed with the client.

- 10.4.6.2 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 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 should 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 should be used for quantitation.

10.4.7.1 If an analyte in the initial calibration is > 15%, then calibration on a curve must be used. Linear or quadratic curve fits may be used if the compound has historically exhibited a nonlinear response. The analyst should consider instrument maintenance to improve the linearity of response. Use of $1/\text{Concentration}^2$ 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 5 points are maintained and the quantitation range is adjusted accordingly. If the % RSD is still >15%, a quadratic or linear curve may 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 Ohio VAP work, the low standard must be at or below the reporting limit.

10.4.7.2 **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 If time remains in the 12-hour period initiated by the DFTPP injection before the initial calibration, samples may be analyzed. Otherwise, proceed to continuing calibration.

10.4.9 Quantitation is performed using the calibration curve or average response factor from the initial curve, not the continuing calibration.

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 6 compounds $>50\%$.

10.6 Continuing Calibration

- 10.6.1 At the start of each 12-hour period, the GC/MS tuning standard must be analyzed. The injection of DFTPP must result in a mass spectrum for DFTPP which meets the criteria given in Table 6.

- 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 The following criteria must be met for the continuing calibration to be acceptable:

- The SPCC compounds must have a response factor of ≥ 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 (4) 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 rules require that any sample with internal standard outliers be reanalyzed undiluted unless a matrix effect is observed. The criteria for acceptance is between 50% and 200% of 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.

- 10.6.4. 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 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.

11.2 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 All samples must be analyzed 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.

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) may 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 should 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, the sample must be reanalyzed at a dilution targeted to bring the largest hit above 50% of the calibration range.

11.3.1 Guidance for Dilutions Due to Matrix

11.3.1.1 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 reanalyzed at a more concentrated dilution. This requirement is approximate and subject to analyst judgement. For example, samples containing organic acids may need to be analyzed at a higher dilution to avoid destroying the column.

11.3.2 Reporting Dilutions

11.3.2.1 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 $4 \pm 2^{\circ}\text{C}$, 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. Reanalysis 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 that 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 shall be completely documented using a Nonconformance Memo and approved by a Technical Specialist and QA Manager. The Nonconformance Memo shall 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 twelve 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%) should 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 should agree to within $\pm 30\%$ 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

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 will 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 shall 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) should be present in the sample spectrum.

12.3.2 The relative intensities of the major ions should agree within $\pm 20\%$. (Example: For an ion with an abundance of 50% in the standard spectrum, the corresponding sample ion abundance should be between 30% and 70%.)

12.3.3 Molecular ions present in the reference spectrum should be present in the sample spectrum.

12.3.4 Ions present in the sample spectrum, but not in the reference spectrum, should be reviewed for possible background contamination or presence of coeluting 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 coeluting 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 was 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.

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

$$\%RSD = \frac{SD}{RF} \times 100$$

RF = Mean of RFs from initial calibration for a compound

SD = Standard deviation of RFs from initial calibration for a compound,

$$= \sqrt{\sum_{i=1}^N \frac{(RF_i - \overline{RF})^2}{N - 1}}$$

RF_i = 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.

$$C_{ex} = \frac{R_x C_{is}}{R_{is} RF}$$

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

$$C_{ex} = A + B \left(\frac{R_x C_{is}}{R_{is}} \right) + C \left(\frac{R_x C_{is}}{R_{is}} \right)^2$$

Where: C = Curvature

12.5.4 The concentration in the sample is then calculated.

12.5.4.1 Aqueous Calculation

$$\text{Concentration, mg / L} = \frac{C_{ex} V_t}{V_o}$$

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):

$$\text{Concentration, mg / kg} = \frac{C_{ex} V_t}{W_s D}$$

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

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-0021.

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 Non-standard analytes

13.4 Training Qualification

13.4.1 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.

13.4.2 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 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 Safety Manual 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 an annual refresher training.
- 15.3 Waste Streams Produced by the Method
 - 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
- 16.2 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-0018
 - 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-0016
 - 16.2.6 Standard and Reagents, NC-QA-017
 - 16.2.7 Acceptable Manual Integration Practices, CA-Q-S-002
 - 16.2.8 Calibration Curves (General), CA-Q-S-005

16.2.9 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

Table 1 - TestAmerica North Canton Primary Standard and 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	2	100	
2,4,5-Trichlorophenol	95-95-4	10	330	1	150	0.02
2,4,6-Trichlorophenol	88-06-2	10	330	1	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	1	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	330	
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	
Benzoic acid	65-85-0	50	1600	10	660	

Table 1 (Cont'd)

Analytes	CAS Number	Water µg/L	Soil µg/kg	Low Level Water, µg/L	Low Level Soil, µg/kg	TCLP mg/L
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	1	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	50	1600	10	330	0.02
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	50	1600	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 2,2'-oxybis(1-chloropropane) was formerly known as bis(2-chloroisopropyl)ether.

Table 2 - TestAmerica North Canton Appendix IX¹ Standard Reporting Limits

Analytes	CAS No.	Water µg/L	Soil µg/kg	Low Level Water, µg/L	Low Level Soil, µg/kg
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
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

Table 2 (Cont'd)

Analytes	CAS No.	Water µg/L	Soil µg/kg	Low Level Water, µg/L	Low Level Soil, µg/kg
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
1,4-Dinitrobenzene	100-25-4	10	330	2	100

¹ The Appendix IX standard contains additional analytes required for the Appendix IX list. The TestAmerica North Canton primary standard must also be analyzed to include all of the Appendix IX list

Table 2A – TestAmerica North Canton Michigan Program¹

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
Atrazine	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 ether	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

Table 2A (Cont'd)

Semivolatile	CAS Number	Michigan Reporting Limits	
		Aqueous mg/L	Low Soil/Sediment mg/kg
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
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
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-Methylnaphthalene	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

Table 2A (Cont'd)

Semivolatile	CAS Number	Michigan Reporting Limits	
		Aqueous mg/L	Low Soil/Sediment mg/kg
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 - Reportable Analytes for TestAmerica North Canton Standard Tests,
Primary Standard**

Analyte	CAS Number	TCLP	TCL	Appendix IX
Pyridine	110-86-1	X		X
N-nitrosodimethylamine	62-75-9			X
Aniline	62-53-3			X
Phenol	108-95-2		X	X
Bis(2-chloroethyl)ether	111-44-4		X	X
2-Chlorophenol	95-57-8		X	X
1,3-Dichlorobenzene	541-73-1		X	X
1,4-Dichlorobenzene	106-46-7	X	X	X
Benzyl alcohol	100-51-6			X
1,2-Dichlorobenzene	95-50-1		X	X
2-Methylphenol	95-48-7	X	X	X
2,2'-oxybis(1-chloropropane)	180-60-1		X	X
4-Methylphenol	106-44-5	X	X	X
N-Nitroso-di-n-propylamine	621-64-7		X	X
Hexachloroethane	67-72-1	X	X	X
Nitrobenzene	98-95-3	X	X	X
Isophorone	78-59-1		X	X
2-Nitrophenol	88-75-5		X	X
2,4-Dimethylphenol	105-67-9		X	X
Benzoic acid	65-85-0		X	
Bis(2-chloroethoxy)methane	111-91-1		X	X
2,4-Dichlorophenol	120-83-2		X	X
1,2,4-Trichlorobenzene	120-82-1		X	X
Naphthalene	91-20-3		X	X
4-Chloroaniline	106-47-8		X	X

Table 3 (Cont'd)

Analyte	CAS Number	TCLP	TCL	Appendix IX
Hexachlorobutadiene	87-68-3	X	X	X
4-Chloro-3-methylphenol	59-50-7		X	X
2-Methylnaphthalene	91-57-6		X	X
Hexachlorocyclopentadiene	77-47-4		X	X
2,4,6-Trichlorophenol	88-06-2	X	X	X
2,4,5-Trichlorophenol	95-95-4	X	X	X
2-Chloronaphthalene	91-58-7		X	X
2-Nitroaniline	88-74-4		X	X
Dimethyl phthalate	131-11-3		X	X
Acenaphthylene	208-96-8		X	X
3-Nitroaniline	99-09-2		X	X
Acenaphthene	83-32-9		X	X
2,4-Dinitrophenol	51-28-5		X	X
4-Nitrophenol	100-02-7		X	X
Dibenzofuran	132-64-9		X	X
2,4-Dinitrotoluene	121-14-2	X	X	X
2,6-Dinitrotoluene	606-20-2		X	X
Diethylphthalate	84-66-2		X	X
4-Chlorophenyl phenyl ether	7005-72-3		X	X
Fluorene	86-73-7		X	X
4-Nitroaniline	100-01-6		X	X
4,6-Dinitro-2-methylphenol	534-52-1		X	X
N-Nitrosodiphenylamine	86-30-6		X	X
Azobenzene ¹	103-33-3		X	
4-Bromophenyl phenyl ether	101-55-3		X	X
Hexachlorobenzene	118-74-1	X	X	X
Pentachlorophenol	87-86-5	X	X	X
Phenanthrene	85-01-8		X	X
Anthracene	120-12-7		X	X
Carbazole	86-74-8		X	
Fluoranthene	206-44-0		X	X
Benzidine	92-87-5		X	
Pyrene	129-00-0		X	X
Butyl benzyl phthalate	85-68-7		X	X
3,3'-Dichlorobenzidine	91-94-1		X	X
Benzo(a)anthracene	56-55-3		X	X
Bis(2-ethylhexyl)phthalate	117-81-7		X	X
Chrysene	218-01-9		X	X
Di-n-octylphthalate	117-84-0		X	X
Benzo(b)fluoranthene	205-99-2		X	X
Benzo(k)fluoranthene	207-08-9		X	X
Benzo(a)pyrene	50-32-8		X	X
Indeno(1,2,3-cd)pyrene	193-39-5		X	X
Dibenz(a,h)anthracene	53-70-3		X	X

Table 3 (Cont'd)

Analyte	CAS Number	TCLP	TCL	Appendix IX
Benzo(g,h,i)perylene	191-24-2		X	X
Benzaldehyde	100-52-7		X	
Caprolactam	105-60-2		X	
1,1-Biphenyl	92-52-4		X	
Atrazine	1912-24-9		X	

¹ Azobenzene is formed by decomposition of 1,2-diphenylhydrazine. If 1,2-diphenylhydrazine is requested, it will be analyzed as azobenzene.

**Table 4 - Reportable Analytes for TestAmerica North Canton Standard Tests,
Appendix IX Standard**

Semivolatiles	CAS Number	TCL	Appendix IX
2-Picoline	109-06-8		X
N-Nitrosomethylethylamine	10595-95-6		X
Methyl methanesulfonate	66-27-3		X
N-Nitrosodiethylamine	55-18-5		X
Ethyl methanesulfonate	62-50-0		X
Pentachloroethane	76-01-7		X
Acetophenone	98-86-2	X	X
N-Nitrosopyrrolidine	930-55-2		X
N-Nitrosomorpholine	59-89-2		X
o-Toluidine	95-53-4		X
3-Methylphenol	108-39-4		X
N-Nitrosopiperidine	100-75-4		X
o,o,o-Triethyl-Phosphorothioate	126-68-1		X
a,a-Dimethyl-phenethylamine	122-09-8		X
2,6-Dichlorophenol	87-65-0		X
Hexachloropropene	1888-71-7		X
p-Phenylenediamine	106-50-3		X
n-Nitrosodi-n-butylamine	924-16-3		X
Safrole	94-59-7		X
1,2,4,5-Tetrachlorobenzene	95-94-3		X
Isosafrole	120-58-1		X
1,4-Dinitrobenzene	100-25-4		X
1,4-Naphthoquinone	130-15-4		X
1,3-Dinitrobenzene	99-65-0		X
Pentachlorobenzene	608-93-5		X
1-Naphthylamine	134-32-7		X
2-Naphthylamine	91-59-8		X
2,3,4,6-Tetrachlorophenol	58-90-2		X

Table 4 (Cont'd)

Semivolatiles	CAS Number	TCL	Appendix IX
5-Nitro-o-toluidine	99-55-8		X
Thionazin	297-97-2		X
1,3,5-Trinitrobenzene	99-35-4		X
Sulfotepp	3689-24-5		X
Phorate	298-02-2		X
Phenacetin	62-44-2		X
Diallate	2303-16-4		X
Dimethoate	60-51-5		X
4-Aminobiphenyl	92-67-1		X
Pentachloronitrobenzene	82-68-8		X
Pronamide	23950-58-5		X
Disulfoton	298-04-4		X
2-secbutyl-4,6-dinitrophenol (Dinoseb)	88-85-7		X
4-Nitroquinoline-1-oxide	56-57-5		X
Famphur	52-85-7		X
Methapyrilene	91-80-5		X
Aramite	140-57-8		X
p-(Dimethylamino)azobenzene	60-11-7		X
p-Chlorobenzilate	510-15-6		X
3,3'-Dimethylbenzidine	119-93-7		X
2-Acetylaminofluorene	53-96-3		X
Dibenz(a,j)acridine ¹	224-42-0		
7,12-Dimethylbenz(a)anthracene	57-97-6		X
3-Methylcholanthrene	56-49-5		X
Hexachlorophene ²	70-30-4		X
Diphenylamine ³	122-39-4		X

¹ Skinner List Compound

² 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)

³ 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 5 - Suggested Instrumental Conditions

Mass Range	35-500 amu
Scan Time	≤1 second/scan
Initial Column Temperature/Hold Time	45°C for 1 minutes
Column Temperature Program	45- 100°C at 25°C/min for 0 min 100 - 280°C at 30°C/min for 0 min 280 - 100°C at 25°C/min for 2 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

Table 6 - DFTPP Key Ions and Ion Abundance Criteria

Mass	Ion Abundance Criteria
51	30 – 60% of mass 198
68	<2% of mass 69
70	<2% of mass 69
127	40 – 60% 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	>1% of mass 198
441	Present, but less than mass 443
442	>40% of mass 198
443	17 – 23% of mass 442

Table 7 - Analytes in Approximate Retention Time Order and Characteristic Ions, Primary Standard

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

**Table 7 - Analytes in Approximate Retention Time Order and Characteristic Ions,
Primary Standard**

Analyte	Primary	Secondary	Tertiary
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

Table 7 (Cont'd)

Analyte	Primary	Secondary	Tertiary
2-Nitroaniline	65	92	138
Dimethylphthalate	163	194	164
Acenaphthylene	152	151	153
2,6-Dinitrotoluene	165	63	89
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

Table 8 - Additional Appendix IX Analytes in Approximate Retention Time Order and Characteristic Ions			
Analyte	Primary	Secondary	Tertiary
2-Picoline	93	66	92
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

Table 8 (Cont'd)

Analyte	Primary	Secondary	Tertiary
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
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 9 - 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 9A - 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
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

Table 9A (Cont'd)

Table 9A 8270C All Analyte Spike Mix	
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
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

Table 9A (Cont'd)

Table 9A 8270C All Analyte Spike Mix	
2,3,5,6-Tetrachlorophenol	100
2,4,5-Trichlorophenol	100
N-Nitrosodimethylamine	100
N-Nitrosodiphenylamine	100

Table 10 - 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
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 11 - 8270C Surrogate Compounds

Surrogate Compounds	Spiking Level, Conc. Added = 20 ug/L / 30 ug/L
Nitrobenzene-d5	20
2-Fluorobiphenyl	20
Terphenyl-d14	20
1,2-Dichlorobenzene-d4 ¹	20
Phenol-d5	30
2-Fluorophenol	30
2,4,6-Tribromophenol	30
2-Chlorophenol-d4 ¹	30

Recovery limits for surrogates are generated from historical data and are maintained by the QA department.

Table 12 - Calibration Ranges, µg/mL

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
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

Table 12 (Cont'd)

Analyte	Calibration Range
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
4-Bromophenyl phenyl ether	0.25-12.5 ug/mL
Hexachlorobenzene	0.25-12.5 ug/mL
Pentachlorophenol	0.25-12.5 ug/mL

Table 12 (Cont'd)

Analyte	Calibration Range
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
Benidine	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
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

¹ 2,2'-oxybis(1-chloropropane) was formerly known as bis(2-chloroisopropyl)ether

² Azobenzene is formed by decomposition of 1,2-diphenylhydrazine. If 1,2-diphenylhydrazine is requested, it will be analyzed as azobenzene.

Note: Nine calibration 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.

Table 13 - Calibration Ranges, Appendix IX, µg/mL

Semivolatiles	Calibration Range
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
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

Table 13 (Cont'd)

Semivolatiles	Calibration Range
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
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

Note: Nine calibrations standards are prepared varying in concentration from 0.05 ug/mL to 12.5 ug/mL. A minimum of five calibration concentrations will be used for initial calibration . The concentration range of each analyte is listed in the table.

Table 14A - 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 14B – 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

**Title: DETERMINATION OF VOLATILE ORGANICS BY GC/MS BASED
ON METHODS 8260B AND 8260A**

[EPA Methods 8260B AND 8260A]

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This SOP was previously identified as SOP No. CORP-MS-0002NC, Rev 2.5, dated 04/03/07

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TABLE OF CONTENTS

1. SCOPE AND APPLICATION 4

2. SUMMARY OF METHOD..... 4

3. DEFINITIONS 5

4. INTERFERENCES 6

5. SAFETY 7

6. EQUIPMENT AND SUPPLIES 9

7. REAGENTS AND STANDARDS 11

8. SAMPLE COLLECTION, PRESERVATION AND STORAGE 12

9. QUALITY CONTROL..... 19

10. CALIBRATION AND STANDARDIZATION..... 22

11. PROCEDURE 26

12. DATA ANALYSIS AND CALCULATIONS..... 31

13. METHOD PERFORMANCE 38

14. POLLUTION PREVENTION 38

15. WASTE MANAGEMENT 38

16. REFERENCES 38

17. MISCELLANEOUS 41

LIST OF TABLES

Table 1	TestAmerica Primary Standard and Reporting Limits
Table 2	TestAmerica Primary Standard Calibration Levels
Table 2A	TestAmerica Primary Standard Calibration Levels- Low Level
Table 3	TestAmerica Appendix IX Standard and Reporting Limits
Table 4	TestAmerica Appendix IX Standard Calibration Levels
Table 5	Reportable Analytes for TestAmerica Standard Tests, Primary Standard
Table 6	Reportable Analytes for TestAmerica Standard Tests, Appendix IX Standard
Table 7	Internal Standards
Table 8	Surrogate Standards
Table 9	Matrix Spike / LCS Control Standards
Table 10	BFB Tune Criteria
Table 11	SPCC Compounds and Minimum Response Factors
Table 12	CCC Compounds
Table 13	Characteristic Ions

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 purged 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 sorbent column where the volatile components are trapped. After purging is completed, the sorbent 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.

- 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 should 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. The use of ultra high purity gases, pre-purged 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.

- 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 should 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 is diluted.

5. SAFETY

- 5.1 Employees must abide by the policies and procedures in the Corporate 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 will be removed and discarded; other gloves will 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 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
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**; therefore, unless they are known to be non-hazardous, all samples should be opened, transferred, and prepared in a fume hood, or under other means of mechanical ventilation where possible. 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 will be kept closed unless transfers are being made.
- 5.6. The preparation of standards and reagents will be conducted in a fume hood with the sash closed as far as the operations will permit.
- 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 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, 0.006 inch ID needle.
- 6.2. Syringe: 5 or 25 mL glass with luerlok tip, if applicable to the purging device.
- 6.3. Balance: Analytical, capable of accurately weighing 0.0001 g, and a top-loading balance capable of weighing 0.1 g
- 6.4. Glassware:
 - 6.4.1. Vials: 20 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 Vocab 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 should be capable of rapidly heating the trap to 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 most recent release of the NIST/EPA mass spectral library should 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 9.4) 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.

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 manufacturers expiration date.

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/ μ L of 4-Bromofluorobenzene in methanol is prepared as described in Sections 7.2.1.1 and 7.2.1.2.

8. SAMPLE COLLECTION, PRESERVATION, AND STORAGE

8.1. Holding times for all volatile analysis are 14 days from sample collection.

8.2. For DoD samples, water samples are normally preserved at $\text{pH} \leq 2$ with 1:1 hydrochloric acid. Unpreserved water samples must be analyzed within seven days.

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. 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 5 g or 25 g 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 $\mu\text{g/kg}$ for most analytes) then it will be necessary to use two additional 5 g EnCore™ samplers or to use field preservation.

- 8.5. Sample collection for medium level analysis using EnCore™ samplers.
 - 8.5.1. Ship one 5 g (or 25 g) 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 25 g) sample into a tared VOA vial containing 5 mL methanol (25 mL methanol for the 25 g 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 $\mu\text{g/mL}$ solution for a nominal 25 g sample, 5 μL for a nominal 5 g sample.) Refer to Section 17.5 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 $\mu\text{g/mL}$ solution for a nominal 25 g sample, 100 μL for a nominal 5 g sample.) Reduce the volume of methanol added to ensure the final volume is 25 mL for nominal 25 g sample or 5 mL methanol for a nominal 5 g sample. Refer to Section 17.4 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 17.4 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 5 g 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 μ L of 2500 μ g/mL solution for a nominal 25 g sample, 5 μ L for a nominal 5 g sample.) Refer to Section 17.5 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 μ L of 50 μ g/mL solution for a nominal 25 g sample, 100 μ L for a nominal 5 g sample.) Reduce the volume of methanol added to ensure the final volume is 25 mL for nominal 25 g sample or 5 mL methanol for a nominal 5 g sample. Refer to Section 17.5 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 μ L of spike to 25 mL methanol or 100 μ L spike to 5 mL methanol). Refer to Section 17.5 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 μ g/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 1 g 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*

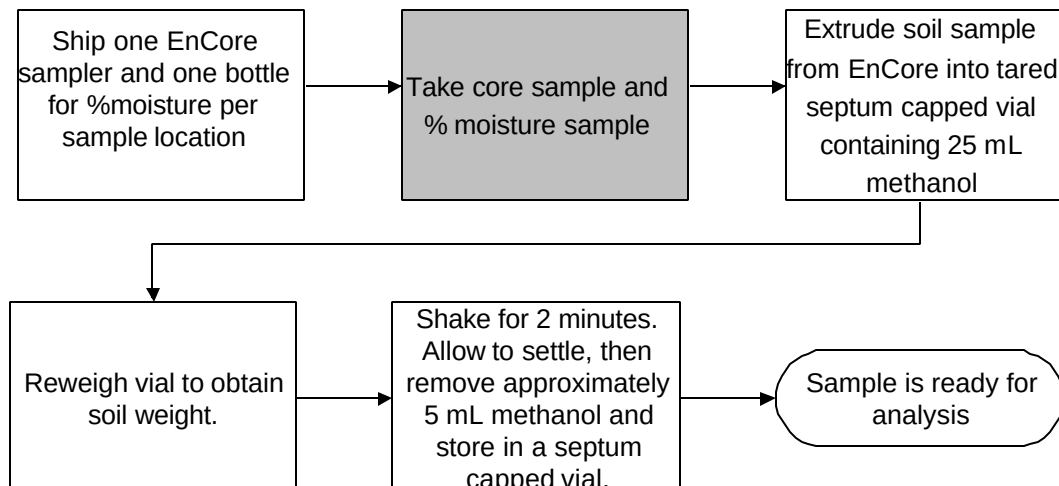
8.8.1. *At specific client request, unpreserved soils packed into glass jars or brass tubes may be accepted and sub-sampled in the lab. This is the old procedure based on method 5030A and method 8260A. It is no longer included in SW846 and is likely to generate results that are biased low, possibly by 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.

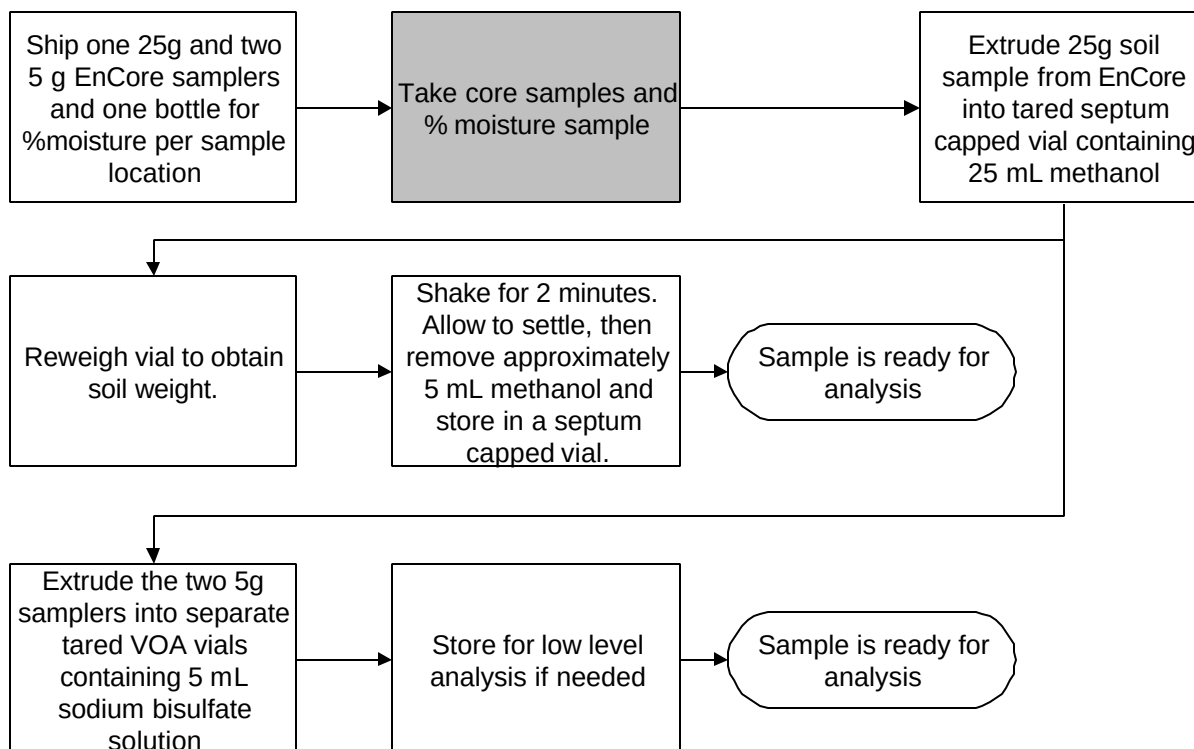
8.10. The maximum holding time is 14 days from sampling until the sample is analyzed. (Samples that are found to be unpreserved still have a 14-day holding time. However they should be analyzed as soon as possible. The lack of preservation must be addressed in the case narrative). Maximum holding time 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.

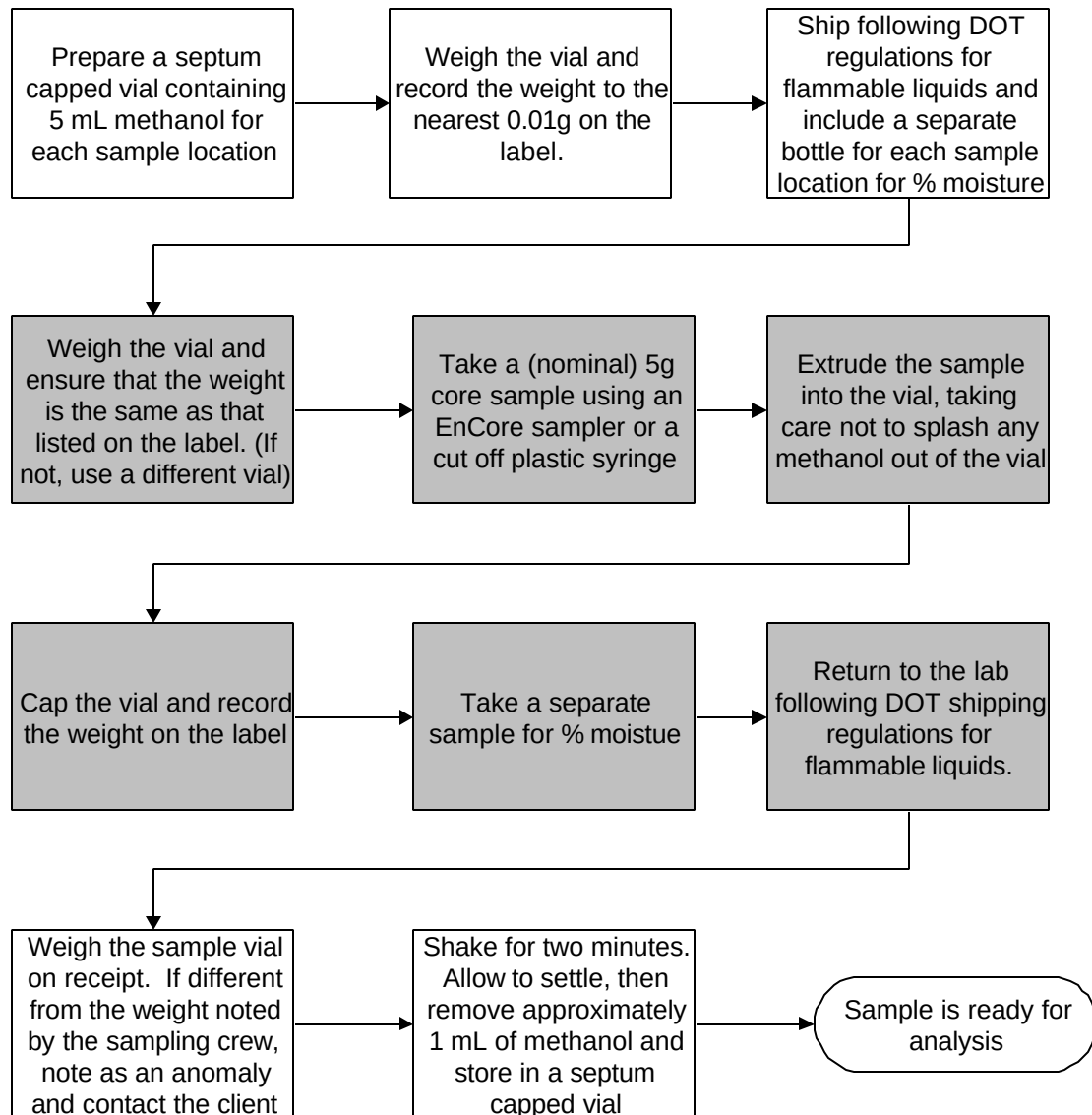
EnCore procedure when low level is not required (field steps in gray)



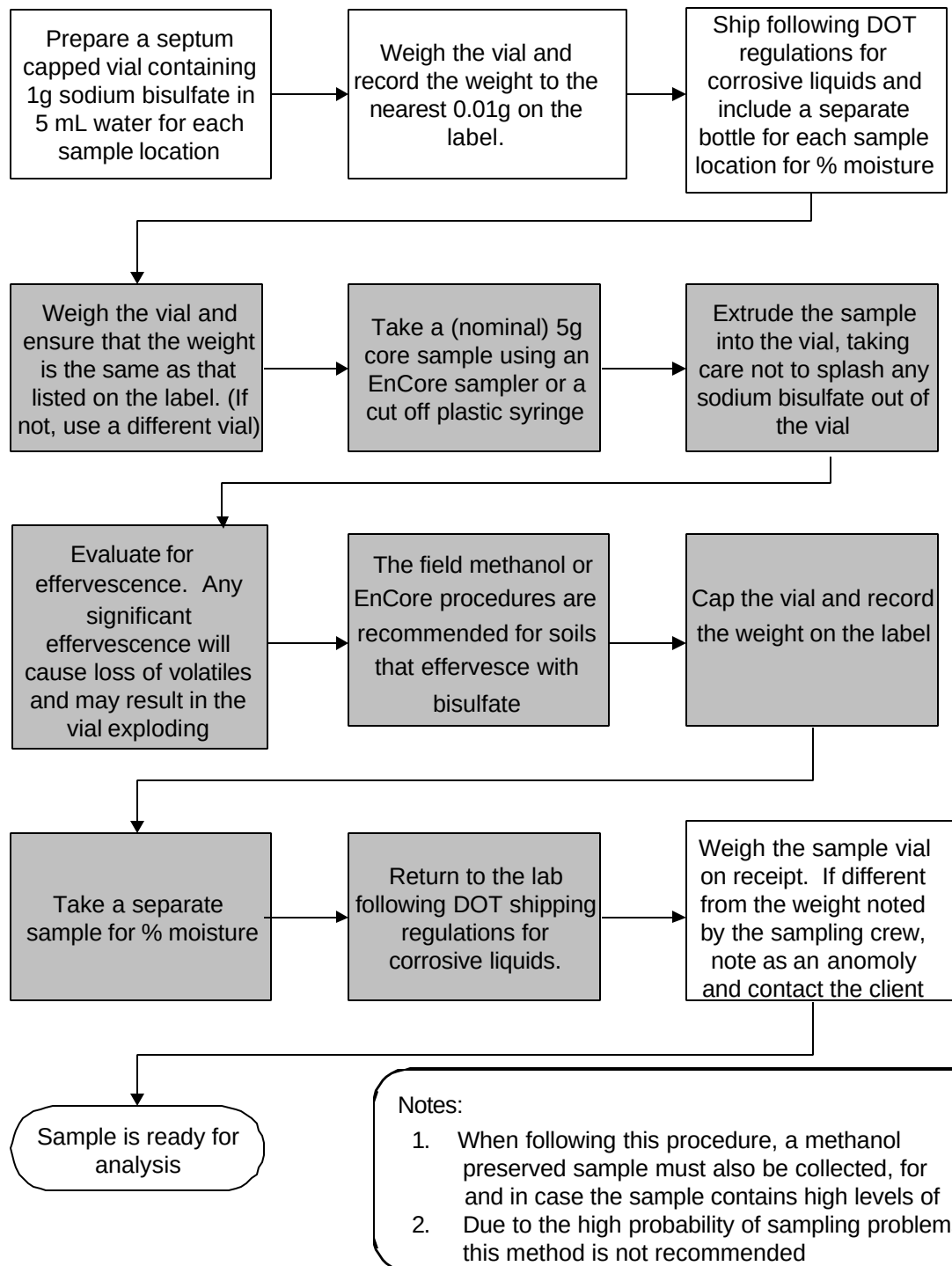
EnCore procedure when low level is required



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 reanalyze if either of the above checks reveal a problem.
- Re-prepare and re-analyze the sample if there is sufficient volume. If there is insufficient volume, the surrogate is narrated.

It is only necessary to re-prepare/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 reparation is not necessary. If the sample is out of control and the MS and/or MSD is in control, then reanalysis or flagging of the data is required. For Ohio VAP samples, all surrogates must be in control, or samples must be reprepared and reanalyzed.

9.2.3. 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. All samples associated with an unacceptable blank will be reprepared and reanalyzed.
- 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.
 - Reanalysis 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 should take place. For Ohio VAP samples, all surrogates must be in control, or reparation 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.

9.4. Laboratory Control Samples (LCS)

9.4.1. For each batch of samples, analyze a 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 re-preparation and reanalysis of the batch. For Ohio VAP samples, all surrogates must be in control on the LCS, or re-preparation 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 should be negotiated with the client. n-Hexane must be spiked and reported for the LCS.

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. n-Hexane must be spiked and reported for the MS/MSD.

- 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.

- 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 reanalysis of the batch.
- If a MS/MSD is not possible due to limited sample, then a LCS duplicate should be analyzed. RPD of the LCS and LCSD are 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-Bromofluorobenzene (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: 15 mL/minute
Make-up Gas Flow:	25–30 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. Refer to TestAmerica North Canton Quality Assurance Manual (NC-QAM-001) for details passing 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 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 on the Agilent 5973 instruments to use 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, 40µg/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 should 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 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.

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 should 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 has historically exhibited a non-linear response. The analyst should 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 5 points are maintained (6 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 5-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 1 g 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) reanalyzed for quantitation. However, if the analyte is not detected, the non-detect may be reported and no further action is necessary.

Note: This procedure may 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 $\leq 20\%$. The recovery for non-CCC compounds must be $\leq 50\%$ with an allowance of up to six compounds $> 50\%$.

- 10.5. Continuing Calibration: The initial calibration must be verified every twelve 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 Sections 10.5.3 and 10.5.4, 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 shall be completely documented using a Non-Conformance Memo and approved by a Supervisor or Group Leader and QA Manager The Non-Conformance Memo shall 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. This is not applicable for Oho 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 a MS/MSD, a 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 reanalyze batch QC with re-analyses of samples. However, any reruns must be as part of a valid batch.

11.3.3 Dilutions should 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.

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 Section 8.5 or 8.6) to the syringe. Add internal standard (if used). Load the sample onto the purge and trap device and analyze as for aqueous samples. If less than 1 μL of methanolic extract is to be added to the water, dilute the methanolic extract such that a volume greater than 1 μL will be added to the water in the syringe. Refer to Section 17.5 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 0.1 gram.

11.5.2 Quickly add 4 mL of methanol, then add 5 μL of a 2500 $\mu\text{g/mL}$ surrogate spiking solution to bring the final volume to 5 mL. Cap the vial and shake for 2 minutes to mix thoroughly. For a MS/MSD or LCS, 4.9 mL of methanol, 5 μL of a 2500 $\mu\text{g/mL}$ 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 Section 8.5 or 8.6) to the syringe. Add internal standard (if used). Load the sample onto the purge and trap device and analyze as for aqueous samples. If less than 5 μL of methanolic extract is to be added to the water, dilute the methanolic extract such that a volume greater than 1 μL 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 should 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 (1 g 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 $\geq 5 \mu\text{L}$. 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 $\mu\text{g/L}$ 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 that TCLP reporting limits will be five times higher than the corresponding aqueous limits).
- 11.7.4. Purge the sample for 11 minutes (the 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, Method 8260A, 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 5 g (or other appropriate aliquot) of sample into a 40 mL vial. Record the weight to the nearest 0.1 g. If method sensitivity is demonstrated, a smaller aliquot may be used. Do not use aliquots less than 0.5 g. If the sample is contaminated with analytes such that a purge amount less than 0.5 g 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. *Connect the purge vessel to the purge and trap device.*

11.8.4. *Rinse a 5 mL gas-tight syringe with organic free water, and fill. Compress to 5 mL. Add surrogate/internal standard (and matrix spike solutions if required.). Add directly to the sample from 11.5.1.*

11.8.5. *The above steps should be performed rapidly and without interruption to avoid loss of volatile organics.*

11.8.6. *Add the heater jacket or other heating device and start the purge and trap unit.*

11.8.7. *Soil samples that have low IS recovery when analyzed (<50%) should be reanalyzed once to confirm matrix effect.*

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 Gently mix the contents of the sample container with a narrow metal or wood spatula. 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 5 μ L of 2500 μ g/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, 5 μ L 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. Reanalysis 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. Reanalysis of samples analyzed while the system was malfunctioning is required. Reanalysis 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 reanalyzed to confirm. If the change in sensitivity is due to instrumental problems all affected samples must be reanalyzed after the problem is corrected. For Ohio VAP projects, the laboratory will reanalyze any sample where the internal standard fails, and there is no evidence of matrix interference.

11.10.3. The surrogate standard recoveries are evaluated to ensure that they are within limits. Corrective action for surrogates out of control will normally be to reanalyze the affected samples. However, if the surrogate standard response is out high and there are no target analytes or tentatively identified compounds, reanalysis may not be necessary. Out of control surrogate standard response may be a matrix effect. It is only necessary to reanalyze a sample once, at the same dilution as the original run, to demonstrate matrix effect, but reanalysis at a dilution could be considered for severe matrix effect.

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 should 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 reanalyzed 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 should be reanalyzed 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. (Note: Care must be taken to ensure that spectral distortion due to co-elution 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 twelve hours as the sample.
- The relative intensities of ions should agree to within $\pm 30\%$ 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 shall 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) should be present in the sample spectrum.

12.2.1.2. The relative intensities of the major ions should 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 should be present in the sample spectrum.

12.2.1.4. Ions present in the sample spectrum but not in the reference spectrum should 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 should 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 should 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 should 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

$$SD = \sqrt{\sum_{i=1}^N \frac{(X_i - X)^2}{N - 1}}$$

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

$$\% \text{ Drift} = \frac{C_{\text{expecte}} - C_{\text{found}}}{C_{\text{expecte}}} \times 100$$

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

$$\text{Concentration mg / L} = \frac{x}{\overline{RF}}$$

12.3.5.3 Calculation of concentration using Linear fit:

Equation 6

$$\text{Concentration mg / L} = A + Bx$$

12.3.5.4. Calculation of concentration using Quadratic fit:

Equation 7

$$\text{Concentration mg / L} = A + Bx + Cx^2$$

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

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)

A_{is} = Area of the characteristic ion for the internal standard

I_s = Amount of internal standard added in ng

Dilution Factor = $D_f = \frac{\text{Total volume purged (mL)}}{\text{Volume of original sample used (mL)}}$

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

A_x , I_s , D_f , A_{is} , same as for water.

V_t = Volume of total extract, mL (Typically 25 mL)

V_a = Volume of extract added for purging, μ L

W_s = Weight of sample extracted, g

$$D = \frac{100 - \% \text{moisture}}{100}$$

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

A_x , I_s , A_{is} , same as for water.

D is as for medium level soils

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:

A_x = Area in the total ion chromatogram for the compound being measured

A_{is} = Area of the total ion chromatogram for the nearest internal standard without interference

$RF = 1$

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

$$\text{Matrix Spike Recovery, \%} = \frac{SSR - SR}{SA} \times 100$$

SSR = Spike sample result

SR = Sample result

SA = Spike added

12.3.7. Relative % Difference calculation for the MS/MSD:

Equation 12

$$RPD = \frac{|\text{MSR} - \text{MSDR}|}{\frac{1}{2}(\text{MSR} + \text{MSDR})} \times 100$$

Where:

RPD = Relative percent difference

MSR = Matrix spike result

$MSDR$ = Matrix spike duplicate result

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 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. This method does not contain any specific modifications that serve to minimize or prevent pollution.

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 Safety Manual 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, Closed System Purge-and-Trap and Extraction for Volatile Organics in Soil and Waste Samples,

Method 5035, Rev 0, December 1996.

16.1.5 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

16.1.6 TestAmerica North Canton Quality Assurance Manual (QAM), current version

TestAmerica Corporate Safety Manual, M-E-0001, 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-0016

16.2.6. Standards and Reagents, NC-QA-017

16.2.7. Laboratory Holding Blanks, NC-QA-0020

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: 12/15/97 (CORP-MS-0002NC)
Revision 2.1: 03/06/00 (CORP-MS-0002NC)
Revision 2.2: 11/28/00 (CORP-MS-0002NC)
Revision 2.3: 05/23/01 (CORP-MS-0002NC)

Revision 2.4: 09/27/04 (CORP-MS-0002NC)
Revision 2.5: 04/03/07 (CORP-MS-0002NC)

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 25 uL of 2500 ug/mL surrogate solution for a nominal 25 g sample).

17.2.2 Modify Sections 8.5.5 and 8.6.9 (add 500 uL of 50 ug/mL spike solution for a nominal 25 g 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 25g 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 levels in water of 0.5 ug/L, 1 ug/L, 5 ug/L, 10 ug/L, 20 ug/L, and 50 ug/L. Some analytes are prepared at higher concentrations.

17.2.5 Samples for Michigan projects frequently require calibration for 2-Methylnaphthalene. Recommended calibration levels for this compounds are 2 ug/L, 10 ug/L, 20 ug/L, 40 ug/L and 80 ug/L.

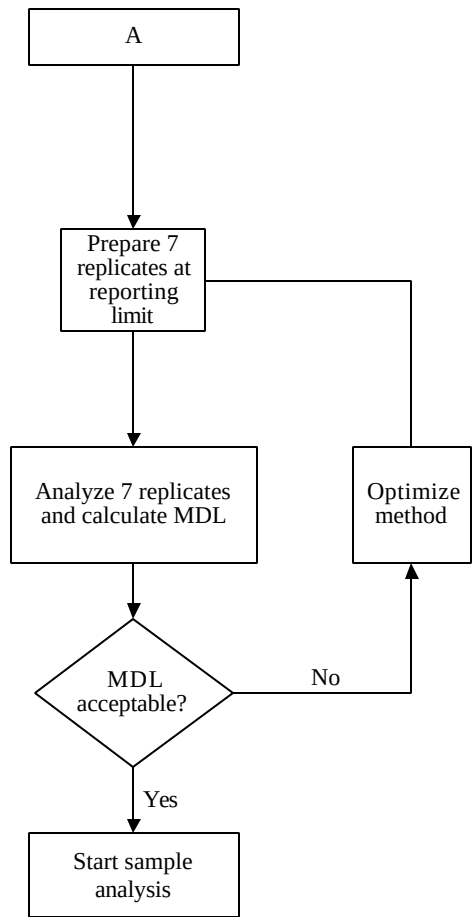
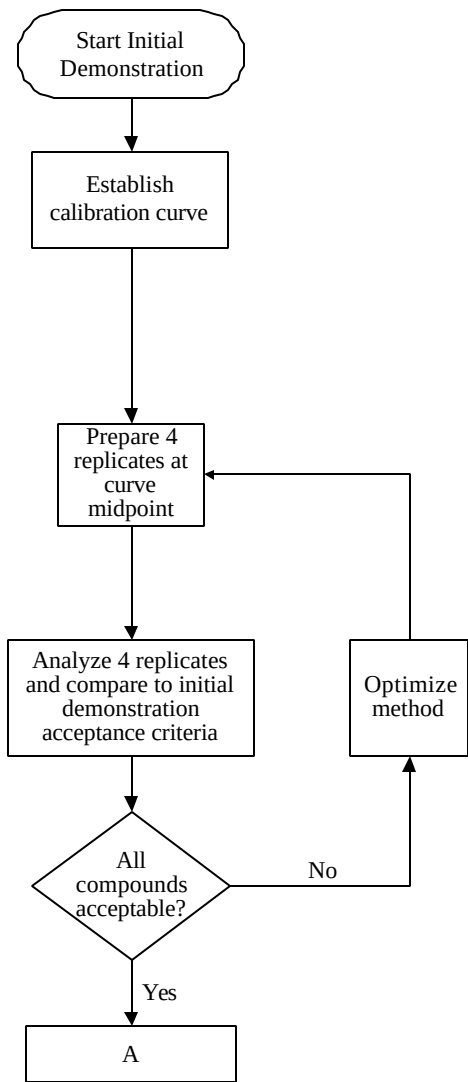


Table 1 - TestAmerica Primary Standard and Reporting Limits

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	1200
Chloromethane	74-87-3	5	1	5	250	1200
Bromomethane	74-83-9	5	1	5	250	1200
Vinyl chloride	75-01-4	5	1	5	250	1200
Chloroethane	75-00-3	5	1	5	250	1200
Trichlorofluoromethane	75-69-4	5	1	5	250	1200
Acrolein	107-02-8	100	20	100	5000	12000
Acetone	67-64-1	20	10	20	1000	2500
Trichlorotrifluoroethane	76-13-1	5	1	5	250	620
Iodomethane	74-88-4	5	1	5	250	620
Carbon disulfide	75-15-0	5	1	5	250	620
Methylene chloride	75-09-2	5	1	5	250	620
tert-Butyl alcohol	75-65-0	200	50	200	10,000	25000
1,1-Dichloroethene	75-35-4	5	1	5	250	620
1,1-Dichloroethane	75-34-3	5	1	5	250	620
trans-1,2-Dichloroethene	156-60-5	5.0	1.0	5.0	250	310
Acrylonitrile	107-13-1	100	20	100	5000	12000
Methyl tert-butyl ether (MTBE)	1634-04-4	20	5	20	1000	2500
Hexane	110-54-3	5	1	5	250	620
cis-1,2-Dichloroethene	156-59-2	5	1	5	250	620
1,2-Dichloroethene (Total)	540-59-0	10	2	10	500	620
Tetrahydrofuran	109-99-9	20	5	20	1000	2500
Chloroform	67-66-3	5	1	5	250	620
1,2-Dichloroethane	107-06-2	5	1	5	250	620
Dibromomethane	74-95-3	5	1	5	250	620
2-Butanone	78-93-3	20	5	20	1000	2500
1,4-Dioxane	123-91-1	500	200	500	25000	62000
1,1,1-Trichloroethane	71-55-6	5	1	5	250	620
Carbon tetrachloride	56-23-5	5	1	5	250	620
Bromodichloromethane	75-27-4	5	1	5	250	620
1,2-Dichloropropane	78-87-5	5	1	5	250	620
cis-1,3-Dichloropropene	10061-01-5	5	1	5	250	620
Trichloroethene	79-01-6	5	1	5	250	620
Dibromochloromethane	124-48-1	5	1	5	250	620
1,2-Dibromoethane	106-93-4	5	1	5	250	620

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 µg/kg	8260A 5030A Med Level Soil µg/kg
1,2,3-Trichloropropane	96-18-4	5	1	5	250	620
1,1,2-Trichloroethane	79-00-5	5	1	5	250	620
Benzene	71-43-2	5	1	5	250	620
Ethylmethacrylate	97-63-2	5	1	5	250	620
trans-1,3-Dichloropropene	10061-02-6	5	1	5	250	620
Bromoform	75-25-2	5	1	5	250	620
4-Methyl-2-pentanone	108-10-1	20	5	20	1000	2500
2-Hexanone	591-78-6	20	5	20	1000	2500
Tetrachloroethene	127-18-4	5	1	5	250	620
Toluene	108-88-3	5	1	5	250	620
1,1,2,2-Tetrachloroethane	79-34-5	5	1	5	250	620
2-Chloroethyl vinyl ether	110-75-8	N/A ²	N/A	50	1000	6200
Vinyl acetate	108-05-4	10	2	10	500	1200
Chlorobenzene	108-90-7	5	1	5	250	620
Ethylbenzene	100-41-4	5	1	5	250	620
Styrene	100-42-5	5	1	5	250	620
t-1,4-Dichloro-2-butene	110-57-6	5	1	5	250	620
m and p Xylenes		10	2	10	500	1200
o-xylene	95-47-6	5.0	1	5	250	620
Total xylenes	1330-20-7	10	2	10	500	1200
1,3-Dichlorobenzene	541-73-1	5	1	5	250	620
1,4-Dichlorobenzene	106-46-7	5	1	5	250	620
1,2-Dichlorobenzene	95-50-1	5	1	5	250	620
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	

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
1,3,5-Trimethylbenzene	108-67-8	5	1	5	250	
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

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
Acetonitrile	50	200	500	1000	2000
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

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
Napthalene	5	20	50	100	200
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
Tetrahydrofuran	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

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
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
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

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
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
Benzene	71-43-2	X	X	X	X
Ethylmethacrylate	97-63-2				X
trans-1,3-Dichloropropene	10061-02-6	X		X	X

Table 5 - Reportable Analytes for TestAmerica Standard Tests, Primary Standard

Compound	CAS Number	TestAmerica Standard List	TCLP	TCL	Appendix IX
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				
sec-Butylbenzene	135-98-8				
4-Isopropyltoluene	99-87-6				
n-Butylbenzene	104-51-8				

Table 5 - Reportable Analytes for TestAmerica Standard Tests, Primary Standard

Compound	CAS Number	TestAmerica Standard List	TCLP	TCL	Appendix IX
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	

Table 7 - Internal Standards

Compound	Standard Concentration µg/mL	Quantitation ion (5 mL purge)
Fluorobenzene	50	96
Chlorobenzene-d5	50	117
1,4-Dichlorobenzene-d4	50	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
1,2-Dichloroethane-d ₄	50
Dibromofluoromethane	50
Toluene-d ₈	50
4-Bromofluorobenzene	50

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.

**Table 9 - Matrix Spike / LCS Control
Compounds**

Compound	Standard Concentration µg /mL
1,1,1-Trichloroethane	50
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
2-Chlorotoluene	50
2-Hexanone	50
4-Chlorotoluene	50
4-Methyl-2-pentanone	50
Acetone	50
Acetonitrile	500
Acrolein	500
Acrylonitrile	500
Benzene	50
Bromobenzene	50
Bromochloromethane	50
Bromodichloromethane	50
Bromoform	50
Bromomethane	50
Carbon disulfide	50
Carbon tetrachloride	50
Chlorobenzene	50

**Table 9 - Matrix Spike / LCS Control
Compounds**

Compound	Standard Concentration µg /mL
Chloroethane	50
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

- 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.

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	<30.0	<20.0
1,1-Dichloroethene	<30.0	<20.0
Chloroform	<30.0	<20.0
1,2-Dichloropropane	<30.0	<20.0
Toluene	<30.0	<20.0
Ethylbenzene	<30.0	<20.0
n-Hexane (Ohio VAP only)	<30.0	<20.0

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

Table 13 - Characteristic Ions

Compound	Primary*	Secondary	Tertiary
cis-1,2-Dichloroethene	96	61	98
2-Butanone	43	72**	
Tetrahydrofuran	42	71	
Chloroform	83	85	47
1,2-Dichloroethane	62	64	98
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

Table 13 - Characteristic Ions

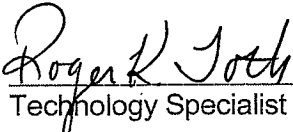
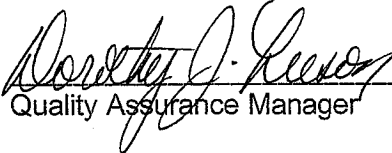
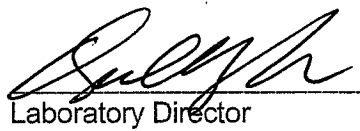
Compound	Primary*	Secondary	Tertiary
Propionitrile	54	52	55
Methacrylonitrile	41	67	52
Isobutanol	41	43	74
Methyl methacrylate	41	69	100
1,1,1,2-Tetrachloroethane	131	133	119
1,2-Dibromo-3-chloropropane	157	155	75
Ethyl ether	59	74	
Ethyl Acetate	43	88	61
2-Nitropropane	41	43	46
Cyclohexanone	55	42	98
Isopropylbenzene	105	120	
Cyclohexane	56	69	84
Methyl Acetate	43	74	
Methyl cyclohexane	83	55	98

* The primary ion should 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.

Title: INDUCTIVELY COUPLED PLASMA – MASS SPECTROMETRY

[EPA Method 200.8 and SW846 Method 6020]

Approvals (Signature/Date):			
	7-9-08		7/10/08
Technology Specialist	Date	Health & Safety Coordinator	Date
	7/9/08		7/11/08
Quality Assurance Manager	Date	Laboratory Director	Date

This SOP was previously identified as SOP No. NC-MT-0002, Rev 4.3, dated 07/28/07

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TABLE OF CONTENTS

1. <i>Scope and Application</i>	3
2. <i>Summary of Method</i>	3
3. <i>Definitions</i>	4
4. <i>Interferences</i>	5
5. <i>SAFETY</i>	6
6. <i>Equipment and Supplies</i>	7
7. <i>Reagents and Standards</i>	8
8. <i>Sample Collection, PRESERVATION and Storage</i>	9
9. <i>Quality Control</i>	9
10. <i>Calibration and Standardization</i>	14
11. <i>Procedure</i>	15
12. <i>Data Analysis and Calculations</i>	17
13. <i>Method Performance</i>	18
14. <i>Pollution Prevention</i>	19
15. <i>Waste Management</i>	19
16. <i>References</i>	20
17. <i>Miscellaneous (Tables, Appendices, Etc.)</i>	21

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 Method 6020 and 200.8. The source method lists fifteen 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 fifteen elements included in the promulgated method.
- 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 CORP-IP-0003NC for preparation details.
- 1.3. The associated QuantIMs 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 quadrupole 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 methods 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. Recommended elemental equations which correct for many of these interferences are listed in Table I. 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), latest version.
- 3.2. *Dissolved Metals* - Those elements which pass through a 0.45 μm membrane filter (sample is acidified after filtration).
- 3.3. *Suspended Metals* - Those elements which are retained by a 0.45 μm membrane filter.
- 3.4. *Total Metals* - The concentration determined on an unfiltered sample following vigorous acid digestion.
- 3.5. *Total Recoverable Metals* - The concentration determined on an unfiltered sample following treatment with hot, dilute mineral acids.
- 3.6. *Instrument Detection Limit (IDL)* - See Section 9.1.1.
- 3.7. *Sensitivity* - The slope of the analytical curve (i.e. the functional relationship between raw instrument signal and the concentration).
- 3.8. *Tuning Solution* - This is a multi-element solution containing analytes which are representative of the entire mass range capable of being scanned by the instrument. It is used to optimize the sensitivity of the instrument and to verify the mass resolution meets method criteria.
- 3.9. *Initial Calibration Verification/Quality Control Standard (ICV/QCS)* - A multi-element standard of known concentrations prepared to verify instrument calibration. This solution must be an independent standard prepared near the mid-point of the calibration curve, and at a concentration other than that used for instrument calibration.
- 3.10. *Continuing Calibration Verification (CCV)*. - A multi-element standard of known concentrations prepared to monitor and verify the instrument daily continuing performance.
- 3.11. *Interference Check Standard (ICS)* - A solution containing both interfering and analyte elements of known concentration that is used to verify background and interelement correction factors.
- 3.12. *Laboratory Control Sample (LCS)* - A multi-element standard of known concentrations which is carried through the entire sample preparation and analysis procedure. This solution is used to verify the accuracy of the sample preparation.

- 3.13. *Reagent Blank* - High purity (> 18 megohm-cm) water carried through the entire digestion process.
- 3.14. *Calibration Blank* - High purity (> 18 megohm-cm) water acidified with the same acid concentrations present in the standards and samples Also referred to as the Initial Calibration Blank (ICB) and Continuing Calibration Blank (CCB).
- 3.15. *Method Detection Limit (MDL)* (see Section 9.1.3)

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. Table II lists isobaric interferences which might possibly affect required analytes. 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 reanalyzed.
 - 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 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.

5. SAFETY

- 5.1. Employees must abide by the policies and procedures in the Corporate 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 will be removed and discarded; other gloves will 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 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
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.

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.
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 should not go near the instrument while in operation.
- 5.5. Exposure to chemicals must be maintained **as low as reasonably achievable**; therefore, unless they are known to be non-hazardous, all samples must be opened, transferred and prepared in a fume hood, or under other means of mechanical ventilation. 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 will be kept closed unless transfers are being made.
- 5.6. It is recommended that neat standards be purchased only as a last resort. The preparation of standards from neat materials and reagents should be conducted in a fume hood with the sash closed as far as the operations will permit.
- 5.7. Standards in solution may be diluted in the open laboratory when syringes and the like are utilized.
- 5.8. The ICP plasma emits strong UV light and is harmful to vision. All analysts must avoid looking directly at the plasma.
- 5.9. 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 or Class A glass volumetric pipettes
- 6.6. Autosampler with autosampler tubes

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. See Table XI.
- 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 XI.
- 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. The use of hydrochloric and sulfuric acids should be minimized for ICPMS without the Collision Cell Technology due to the higher incidence of molecular-ion interferences with the presence of these acids. Excessive amounts of nitric acid can also lead to molecular interferences.
- 7.5. Reagent water
 - 7.5.1. ASTM Type I or equivalent for the elements of interest, generated using an ion-exchange water polishing system.

- 7.6. Rinse Solution. Carefully dilute 200 mL of concentrated HNO_3 and 40mL of concentrated HCl to 4.0 L with reagent water for the Perkin Elmer Elan 6100. Carefully dilute 80 ml of concentrated HNO_3 to 4.0 L with reagent water for the Thermo X Series 2.

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 $4^\circ \pm 2^\circ\text{C}$ until the time of preparation.
- 8.3. The analytical holding times for metals are six months from the time of collection.
- 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 X 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 should 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. $\text{IDL} = (3) (s)$, 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 should 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 contamination level).

Note: For Ohio VAP samples, all analytes must be less than the reporting limit.

9.3.2. Corrective Action for Method Blank

- 9.3.2.1. If the analyte is a common laboratory contaminant (copper, iron, lead, calcium, magnesium, potassium, sodium 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.**

- 9.3.2.2. Repreparation and reanalysis 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 reanalysis 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. On-going 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 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.**
 - 9.4.2.3. Corrective action will be repreparation and reanalysis 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 reparation and reanalysis of the batch.
- 9.5.2.2. If the native analyte concentration in the MS/MSD exceeds 4x 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) should 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 interference's, reparation and reanalysis of the MS/MSD may be necessary.

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. 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 X provides a summary of quality control requirements including frequency, acceptance criteria and corrective action.
- 9.10. 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.
- 9.11. 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 X provides a summary of quality control requirements including frequency, acceptance criteria and corrective action
- 9.12. 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 V. 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" should be monitored for possible interferences.
- 9.12.1. 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 +/- 2x 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.

- 9.13. 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, then perform appropriate dilutions until the internal standard recoveries are within the method criteria. In no case, may Method 6020 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.
- 9.14. Serial Dilution **Method 6020 only.** One serial five-fold dilution should 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.
- 9.15. 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 should be suspected.
- 9.16. General Corrective Action Requirements. The general requirements for evaluation of QC results and corrective action for failures is described in TestAmerica Policy QA-003.
- 9.17. Nonconformance and Corrective Action
- 9.17.1. 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. 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 4 times. For Method 200.8, the tuning solution must be analyzed five times with a relative standard deviation less than 5%.

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. Due to a limitation of the instrument software, the resolution requirement for method 200.8 of 1.0 amu full width at 5% peak height cannot be verified automatically. If the mass resolution requirement of 0.9AMU at 10% peak height is met, the 200.8 requirement is also satisfied.

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.

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 shall be completely documented using a Nonconformance Memo and is approved by a Technical Specialist and QA Manager. The Nonconformance Memo shall 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.

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 CORP-IP-0003NC for preparation 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 reanalyze samples that are more concentrated than the linear range for an analyte or specific isotope of interest. No analyte may be reported from an analysis of a diluted sample in which the analyte concentration is less than 5 times the IDL (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 should 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 an 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 two 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:

$$\%R = 100 \times \left(\frac{\text{Found (ICV)}}{\text{True (ICV)}} \right)$$

12.2. CCV percent recoveries are calculated according to the equation:

$$\%R = 100 \times \left(\frac{\text{Found (CCV)}}{\text{True (CCV)}} \right)$$

12.3. Matrix Spike Recoveries are calculated according to the following equation:

$$\%R = 100 \times \left(\frac{\text{SSR} - \text{SR}}{\text{SA}} \right)$$

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:

$$\text{RPD} = 100 \times \left[\frac{(\text{DU1} - \text{DU2})}{(\text{DU1} + \text{DU2}) / 2} \right]$$

Where:

DU1 = Sample result

DU2 = Sample duplicate result

12.5. The final concentration for an aqueous sample is calculated as follows:

$$\text{Result (ug/L)} = \frac{(C \times V1 \times D)}{V2}$$

Where:

C = Concentration from instrument readout, ppb

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:

$$\text{Result (ug/kg)} = \frac{(C \times V \times D)}{W}$$

Where:

C = Concentration from instrument readout, ppb

D = Instrument dilution factor

V = Final volume in liters after sample preparation

W = Weight, in g, of wet sample digested

12.7. Sample results should be reported according to the following significant figure rules.

12.7.1. All uncorrected values less than the detection limit are reported as "less than" the detection limit.

12.7.2. Positive results for target analytes are reported to three significant figures if the method is used without dilution.

12.8. Positive results obtained after dilution and results for non-certified analytes are reported to two significant figures

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 I 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. Method performance is determined by the analysis of matrix spike and matrix spike duplicate samples as well as method blanks and laboratory control samples. The matrix spike recovery should fall within historical laboratory control limits and the matrix spike duplicates should compare within 20% RPD. Method blanks must meet the criteria specified in Section 9.3. The laboratory control samples should recover within 20% of the true value until in house control limits are established.

13.4. Training Qualifications

13.4.1. 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. This method does not contain any specific modifications that serve to minimize or prevent pollution.

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 Safety Manual 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. Revision History

<u>Historical File:</u>	Revision 0: 08/01/95	Revision 4.1: 10/01/03
	Revision 1: 06/06/01	Revision 4.2: 01/08/04
	Revision 3: 03/26/02	Revision 4.3: 07/28/07
	Revision 4: 03/06/03	

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, CORP-IP-0003NC

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-0016

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 XII 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.

17.2.1.2. The results of the calibration blank as well as all other blanks must be less than the reporting limit, not 3 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.

TABLE I: Recommended Elemental Interference Equations

Element	Isobaric Correction	Mathematical Equation
Al	none	$(1.0000)(27M^*)$
Sb	none	$(1.0000)(121M)$
As	ArCl, Se	$(1.0000)(75M) - (3.1278)(77M) + (1.0177)(78M)$
Ba	none	$(1.0000)(135M)$
Be	none	$(1.0000)(9M)$
Cd	MoO, Sn	$(1.0000)(114M) - (0.0268)(118M) - (1.0000)(135M)$
Ca	none	$(1.0000)(44M)$
Cr	none	$(1.0000)(52M)$
Co	none	$(1.0000)(59M)$
Cu	none	$(1.0000)(65M)$
Fe	none	$(1.0000)(57M)$
Pb	none	$(1.0000)(208M) + (1.0000)(207M) + (1.0000)(206M)$
Mg	none	$(1.0000)(25M)$
Mn	none	$(1.0000)(55M)$
Ni	none	$(1.0000)(60M)$
K	none	$(1.0000)(39M)$
Se	Ar2	$(1.0000)(78M) - (1.1869)(76M)$
Ag	none	$(1.0000)(107M)$
Na	none	$(1.0000)(23M)$
Tl	none	$(1.0000)(205M)$
V	ClO, Cr	$(1.0000)(51M) - (3.1081)(53M) + (0.3524)(52M)$
Zn	none	$(1.0000)(66M)$
6Li	Li (natural)	$(1.0000)(6M) - (0.0813)(7M)$
Sc	none	$(1.0000)(45M)$
Y	none	$(1.0000)(89M)$
Rh	none	$(1.0000)(103M)$
In	Sn	$(1.0000)(115M) - (0.0149)(118M)$
Tb	none	$(1.0000)(159M)$
Ho	none	$(1.0000)(165M)$
Bi	none	$(1.0000)(209M)$

* M = Total ion count rate at the specified mass

TABLE II: Isobaric Molecular-Ion Interferences Which Could Affect the Analytes

Analyte	Interferences						
	Oxygen	Hydroxyl	Nitrogen	Chlorine	Sulfur	Carbon	Other
¹²¹ Sb	PdO		AgN			AgC	
¹²³ Sb	AgO		AgN	SrCl	ZrS	CdC	
⁷⁵ As	CoO	NiOH	NiN	ArCl	CaS	CuC	
¹³⁸ Ba	SnO	SbOH					
¹³⁷ Ba	SbO	SnOH		MoCl			
¹³⁶ Ba	SnO	SnOH				SnC	
¹³⁵ Ba	SnO	SnOH		MoCl			
¹³⁴ Ba	SnO	SnOH	SnN	MoCl		SnC	
¹³² Ba	SnO, CdO	InOH	SnN	MoCl	MoS	SnC	
¹³⁰ Ba	CdO	CdOH	SnN, CdN	MoCl	MoS	SnC	
⁹ Be							
¹¹⁴ Cd	MoO	MoOH	MoN	SeCl	SeS		
¹¹² Cd	MoO, ZrO	MoOH	MoN	AsCl, SeCl	SeS	MoC	
¹¹¹ Cd	MoO	MoOH	MoN	GeCl			
¹¹⁰ Cd	MoO, ZrO		MoN, ZrN	GeCl, AsCl	SeS	MoC	
¹¹³ Cd	MoO	MoOH		SeCl, AsCl			
¹¹⁶ Cd	MoO						
¹⁰⁶ Cd	ZrO		MoN, ZrN		GeS	MoC, ZrC	
¹⁰⁸ Cd	MoO, ZrO	ZrOH	MoN, ZrN	GeCl	SeS, GeS	MoC, ZrC	
⁵² Cr	ArO	ClOH				ArC	
⁵³ Cr	ClO	ArOH	KN	NCl, OCl		KC	
⁵⁰ Cr	SO		ArN		SO	ArC	Mo ⁺⁺
⁵⁴ Cr		ClOH	ArN, CaN			CaC	
⁵⁹ Cr	CaO	CaOH	ScN	MgCl	AlS	TiC	Sn ⁺⁺
⁶³ Cu	TiO, PO ₂	TiOH	TiN	SiCl, MgCl	PS	VC	ArNa
⁶⁵ Cu	TiO	TiOH	VN	SiCl	SS, SO ₂ H	CrC	
²⁰⁸ Pb							
²⁰⁶ Pb							
²⁰⁷ Pb							
²⁰⁴ Pb							
⁵⁵ Mn	KO	ArOH	KN		NaS	CaC	Cd ⁺⁺

TABLE II cont'd: Isobaric Molecular-Ion Interferences Which Could Affect the Analytes

Analyte	Interferences						
	Oxygen	Hydroxyl	Nitrogen	Chlorine	Sulfur	Carbon	Other
²⁰⁰ Hg	WO	WOH	WN				
¹⁹⁹ Hg	WO	WOH					
²⁰¹ Hg		WOH					
¹⁹⁸ Hg	WO	TaOH	WN			WC	
²⁰⁴ Hg							
¹⁹⁶ Hg			WN				
⁵⁸ Ni	CaO	KOH	CaN	NaCl	MgS	TiC	Cd ⁺⁺ , Sn ⁺⁺
⁶⁰ Ni	CaO	CaOH	TiN	MgCl, NaCl	SiS	TiC	Sn ⁺⁺
⁶² Ni	TiO	ScOH	TiN	AlCl, MgCl	SiS	TiC, CrC	Sn ⁺⁺
⁶¹ Ni	SeO	CaOH	TiN	MgCl	SiS	TiC	
⁶⁴ Ni	TiO	TiOH	TiN, CrN	SiCl, AlCl	SS	CrC	
⁸⁰ Se	ZnO	CuOH	ZnN	ScCl, CaCl	TiS	ZnC	
⁷⁸ Se	NiO	NiOH	ZnN	CaCl, KCl	TiS	ZnC	
⁸² Se	ZnO	CuOH	ZnN	TiCl, ScCl	TiS, CrS		
⁷⁶ Se	NiO	CoOH	NiN	KCl	CaS	ZnC	
⁷⁷ Se	NiO	CuN	CuN	CaCl, ArCl	ScS	CuC	
⁷⁴ Se	NiO	NiN	NiN	ClCl, KCl	CaS	NiC	
¹⁰⁷ Ag	ZrO	ZrOH		GeCl	AsS	MoC	
¹⁰⁹ Ag		MoOH	MoN	GeCl	SeS	MoC	
²⁰⁵ Tl							
²⁰³ Tl		WOH					
⁵¹ V	ClO	SOH	ClN	ClO, ClN	FS	KC	
⁵⁰ V	SO		ArN			ArC	Mo
⁶⁴ Zn	TiO	TiOH	TiN, CrN	SiCl, AlCl	SS	CrC	
⁶⁶ Zn	TiO	TiOH	CrN	PCl, SiCl	SS	FeC	
⁶⁸ Zn	CrO	VOH	FeN	PCl	ArS	FeC	Ba ⁺⁺
⁶⁷ Zn	VO	TiOH, Cr	CrN	SCl	ClS	MnC	Ba ⁺⁺
⁷⁰ Zn	FeO	CrOH	GeN	ClCl	ArS	NiC	

Note: The information provided in this table does not indicate that all of the described interferences need to be tested. However, the table can be consulted for informational purposes if unusual samples are encountered.

Table III: Changes in Isobaric Molecular-Ion Interferences with Changing Plasma Conditions**

	Molecular Interference	Nebulizer		
		High	Average	Low
Oxides:	ScO/Sc	0.00326	0.00055	0.00116
	YO/Y	0.00568	0.00395	0.00353
	TbO/Tb	0.0156	0.00648	0.00614
	ClO, Cl	0.00725	0.00227	0.00233
Hydroxides:	ScOH/Sc	0.00040	0.00011	0.00000
	YOH/Y	0.00078	0.00044	0.00048
	TbOH/Tb	0.00034	0.00008	0.00011
	ClOH/Cl	0.00048	0.00031	0.00029
Chlorine:	ClO/Cl	0.00725	0.00227	0.00233
	ClOH/Cl	0.00048	0.00031	0.00029
	ArCl/Cl	0.00605	0.00091	0.00477

** Information for this table is being determined by the EPA.

Table IV: Recommended Internal Standards

Method 6020	Method 200.8
Li	Sc
Sc	Y
Y	In
Rh	Tb
In	Bi
Tb	
Ho	
Bi	
Ge	

Table V: Interference Check Sample Components and Concentrations
(ICSAB minors are suggested spike levels)

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

Table VI: Sample Preservation and Holding Times

Measurement Parameter	Container (1)	Preservative (2)	Maximum Holding Time (3)
<i>Waters:</i>			
Metals (4)	P,G	HNO ₃ to pH < 2	6 months
<i>Soils/Sediments/Wastes:</i>			
The preservation required for soil/sediment/waste samples is maintenance at 4°C (± 2°C) until digestion.			

Footnotes: (1) Polyethylene (P) or glass (G).

(2) Sample preservation is performed by the sampler immediately upon sample collection.

(3) Samples must be analyzed as soon as possible after collection. The times listed are the maximum times that sample may be held before analysis and still considered valid. Holding times are calculated from the date when the sample was collected.

(4) Samples are filtered immediately on-site by the sampler before adding preservative for dissolved elements.

Table VII: Suggested Mass Choices

Boldface masses indicate the masses which must have the most impact on data quality and the elemental equations used to collect the data. Suggested masses for method 200.8 are in “quotes”.

Mass	Element of Interest
“27”	Aluminum
121, “123”	Antimony
“75”	Arsenic
138, “137” , 136, 135 , 134, 132, 130	Barium
“9”	Beryllium
114 , 112, “111” , 110, 113, 116, 106	Cadmium
42, 43, 44 , 46, 48	Calcium
“52” , 53, 50 , 54	Chromium
“59”	Cobalt
“63” , 65	Copper
56 , 54 , 57, 58	Iron
“208” , “207” , “206” , 204	Lead
24, 25, 26	Magnesium
“55”	Manganese
58, “60” , 62, 61 , 64	Nickel
39	Potassium
80, 78, “82” , 76, 77, 74	Selenium
“107” , 109	Silver
23	Sodium
“205” , 203	Thallium
“51” , 50	Vanadium
64, “66” , 68 , 67, 70	Zinc
72	Germanium
139	Lanthanum
118	Tin
35, 37	Chlorine
“98” , 96, 92, 97 , 94	Molybdenum

Note: It is strongly recommended that elements other than those of interest be monitored to indicate other potential molecular interferences which could affect the data quality.

Table VIII: 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.

Method 6020

Element	Concentration (mg/L)
Solution A	
Mg	10
Rh	10
Pb	10
Solution B	
Li	10
Co	10
In	10
Tl	10

Method 200.8

Element	Concentration (mg/L)
Be	10
Mg	10
Co	10
In	10
Pb	10

**Table IX: Suggested Tuning and Response Factor Criteria Minimum Response
from Tuning Solution****With a Peristaltic Pump Speed of 12 RPM****Perkin Elmer Elan 6100**

Be	> 1,000
Mg	> 10,000
Rh	>100,000
Pb	> 50,000
Li	> 2,000
Co	> 20,000
In	> 1,000
Tl	> 1,000

**Minimum Response from Tuning Solution
With a Peristaltic Pump Speed of 40 RPM****Thermo X Series 2**

Be	> 8,000
In	>300,000
Pb	> 10,000
Co	>150,000
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

Table X: Summary of Quality Control Requirements

QC Parameter	Frequency	Acceptance Criteria	Corrective Action
ICV/QCS	Beginning of every analytical run	0 - 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

Table X cont'd: Summary of Quality Control Requirements

QC Parameter	Frequency	Acceptance Criteria	Corrective Action
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

Table XI: ICP/MS Calibration and Calibration Verification Checklist

Perkin Elmer Elan 6100

Suggested Levels in mg/L

Element	Calibration			ICV	CCV
	1	2	3		
Aluminum	50	5000	10000	25	50
Antimony	2	50	100	25	50
Arsenic	2	50	100	25	50
Barium	1	50	100	25	50
Beryllium	1	50	100	25	50
Cadmium	0.5	50	100	25	50
Chromium	2	50	100	25	50
Cobalt	1	50	100	25	50
Copper	2	50	100	25	50
Iron	20	5000	10000	25	50
Lead	1	50	100	25	50
Manganese	1	50	100	25	50
Nickel	2	50	100	25	50
Selenium	2	50	100	25	50
Silver	0.5	50	100	25	50
Thallium	1	50	100	25	50
Vanadium	5	50	100	25	50
Zinc	10	50	100	25	50

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.

Table XII: ICP/MS Calibration and Calibration Verification Checklist

Thermo X Series 2
Suggested Levels in mg/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.

Table XIII: 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

APPENDIX A

OPERATION INSTRUCTIONS – P.E. 6100 **ICP/MS INSTRUCTIONS**

A. Light the plasma and start the peristaltic pump.

1. Allow the instrument to warm up approximately 30 minutes.

B. Daily Performance

1. Open Daily2_asx workplace.
2. Open the sampling tab in the method screen.
3. Select and initialize the autosampler. Click O.K.
4. Click on probe. Click go to rinse. Send probe to tube #8.
5. Allow solution to reach plasma and then click on analyze sample.
6. When analysis is complete, send probe back to rinse.

C. Analysis Setup

1. Open Analysis workspace.
2. Under the method window, open the report tab and type in your report filename. Save the method.
3. Highlight the dataset window. Go to file and select new. Type in the dataset name (normally the same as the filename).
4. Highlight autosampler window. Type in your autosampler locations and samples. Right click under measurement action and select what needs to be analyzed. Right click under method description and select method. Verify that all times and rpms on table are correct. Go to file and click save as. Type autosampler table name (normally the same as the filename).

D. Analyze samples

1. Highlight samples to be analyzed.
2. Left click on analyze batch.

X-Y Adjustment

Adjusts torch to achieve best intensities. This should be done whenever anything is done to the torch or the cones.

1. Open X-Y_asx.wrk.
2. Under the method window, go to the sampling tab and send the probe to tube #8.
3. Allow solution to reach plasma and hit analyze sample.
4. Adjust x and y while watching the signal on the realtime window. Adjust only until signal is at its highest.

NEB Lens

This also is done to get best intensities. It effects the shape and the depth of the plasma. This should be done if your oxides or doubly charged are >3%.

1. Open neb lens power oxides.wrk
2. Under the method window, go to the sampling tab and send the probe to tube #8.
3. Click on the Autooptimize tab. Select nebulizer gas flow.
4. Click get analyte list.
5. Make sure solution has reached the plasma and click optimize.
6. When done, save the optimization file.

Autolens

Each element done represents a section of the mass spectrum. Be is on the low end, CO is in the middle and Pb is on the higher end. If there is a problem with any particular section of the spectrum, it may be an indication that this needs done. This should probably be performed on a weekly basis.

1. Open Autolens_asx.wrk.
2. Under the method window, go to the sampling tab and send the probe to tube #8.
3. Click on the autolens tab.
4. Click on clear calibration.
5. Click on get analyte list.
6. Make sure the solution has reached the plasma and click on calibrate.
7. Save the optimization file.
8. In the interactive window, the optimization curve can be printed.

Dual Detector Calibration

This extends the dynamic range of the detector. This should be done on a weekly basis. All analytes of interest must be included in the solution.

1. Open dual detector2.wrk
2. Under the method window, go to the sampling tab and send the probe to tube #8.
3. Click on dual detector cal tab.
4. Click on clear calibration.
5. Click on get analyte list.
6. Make sure the solution has reached the plasma and click on calibrate.
7. When complete, check to make sure that all r values are at least 0.999.
8. Save the optimization file.
9. In the interactive window, the plots can be displayed.

Tuning

This adjusts the electronics to assure the accuracy of the mass spec. The resolution adjustment assures that the resolution of each mass of interest is within range. This should be done daily.

1. Open tuning_asx.wrk.
2. Under the method window, go to the sampling tab and send the probe to tube #8.
3. Allow sample to reach the plasma and click on tune mass spec.
4. Make sure measured mass and peak width is within range. If its not, resolution will need adjusted. Adding 30 units will decrease the amu by about 0.1. Subtracting 30 units will increase the amu about 0.1.

General Notes

1. All optimizations can be done with the tuning solution, except the dual detector cal needs the cross cal standard with all the elements of interest.
2. All optimizations can be run at 12 rpm, except the dual detector calibration should be at 24 rpm.

APPENDIX B

OPERATION INSTRUCTION – THERMO X SERIES 2 **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 that 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
- 1.2 Perform the daily maintenance as outlined in Appendix C.
- 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 Thermo service agent for advice.

- 1.4 Once the instrument is in the *Operate* state, it should 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 that 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 using *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.

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 >0.020, 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 that 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 should 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 that 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 run. The calibration and QC concentration information is 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 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 that 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 15ml 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 50ml 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, and 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 lightning). 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, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using a Nonconformance Memo and is approved by a Technical Specialist and QA Manager. If contractually required, the client shall be notified. The Nonconformance Memo shall 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.
 - 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 2 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.

APPENDIX C

ICPMS MAINTENANCE SCHEDULE

Daily

Change sample and internal standard pump tubing and pump windings

Check argon gas supply level

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 sample flow for cross flow nebulizer

Check pressure for vacuum systems

Check daily performance

As Needed

Clean Sampler and skimmer cones

Clean plasma torch assembly to remove accumulated deposits

Clean nebulizer

Replace sample and internal standard capillary tubing and autosampler sipper probe

Replace drain tubing

Perform necessary optimizations

Clean autolens

Monthly

Inspect air filters; clean or replace as needed

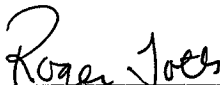
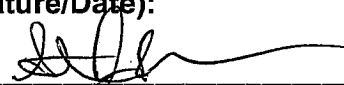
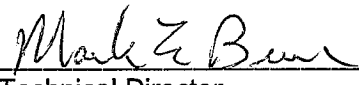
Bi-Yearly

Change oil in vacuum pumps

Check water in coolflow

**Title: PREPARATION AND ANALYSIS OF MERCURY IN SOLID
SAMPLES BY COLD VAPOR ATOMIC ABSORPTION SPECTROSCOPY**

[Method: SW846 7471A]

Approvals (Signature/Date):			
	12-5-07		12-6-07
Technology Specialist	Date	Health & Safety Coordinator	Date
	12/6/07		12/7/07
Quality Assurance Manager	Date	Laboratory Director	Date
	12/6/07		
Technical Director	Date		

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TABLE OF CONTENTS

1. SCOPE AND APPLICATION	3
2. SUMMARY OF METHOD	3
3. DEFINITIONS	3
4. INTERFERENCES	3
5. SAFETY	4
6. EQUIPMENT AND SUPPLIES	7
7. REAGENTS AND STANDARDS	8
8. SAMPLE COLLECTION, PRESERVATION AND STORAGE	9
9. QUALITY CONTROL	9
10. CALIBRATION AND STANDARDIZATION	13
11. PROCEDURE	13
12. DATA ANALYSIS AND CALCULATIONS	17
13. METHOD PERFORMANCE	19
14. POLLUTION PREVENTION	19
15. WASTE MANAGEMENT	19
16. REFERENCES	20
17. MISCELLANEOUS (TABLES, APPENDICES, ETC .)	20

LIST OF APPENDICES

APPENDIX A – TABLES	24
APPENDIX B - TESTAMERICA NORTH CANTON Hg DATA REVIEW CHECKLIST	28
APPENDIX C - MSA GUIDANCE	30
APPENDIX D - TROUBLESHOOTING GUIDE	33
APPENDIX E- CONTAMINATION CONTROL GUIDELINES	35
APPENDIX F - PREVENTATIVE MAINTENANCE	37
APPENDIX G – INSTRUMENT SET-UP	39

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 SW-846 Method 7471A.
- 1.2. The associated LIMs method code is O9.
- 1.3. CVAA analysis provides for the determination of total mercury (organic and inorganic). The combination of the oxidants, potassium permanganate and potassium persulfate, 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 7471A is applicable to the preparation and analysis of mercury in soils, sediments, bottom deposits, wastes, wipes and sludge-type materials. All matrices require sample preparation prior to analysis.
- 1.5. The TestAmerica North Canton reporting limit for mercury in solid matrices is 0.033 mg/kg based on a 0.6 g sample aliquot (wet weight).

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 hydrochloric and nitric acids. Organic mercury compounds are oxidized with potassium permanganate 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. Total Metals: The concentration determined on an unfiltered sample following digestion.

4. INTERFERENCES

Chemical and physical interferences may be encountered when analyzing samples using this method.

- 4.1. Potassium permanganate which is used to breakdown 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.

- 4.2. 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.
- 4.3. Chlorides can cause a positive interference. Samples high in chlorides require additional permanganate (as much as 25 mL) because, during the oxidation step, chlorides are converted to free chlorine, which also absorbs radiation at 253.7 nm. Care must be taken to ensure that free chlorine is absent before the mercury is reduced and swept into the cell. This is accomplished by adding excess hydroxylamine reagent (25 mL) and purging the sample headspace before stannous chloride is added. Both inorganic and organic mercury spikes have been quantitatively recovered from seawater using this technique.

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.

- 4.4. 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.
- 4.5. 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.
- 4.6. 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.

5. SAFETY

- 5.1. Employees must abide by the policies and procedures in the Corporate Safety 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 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
Mercury (1,000 PPM in Reagent)	Oxidizer Corrosive Poison	0.1 Mg/M3 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.
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.

Potassium Permanganate	Oxidizer	5 Mg/M3 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 will be removed and discarded; other gloves will be cleaned immediately.
- 5.5. Exposure to chemicals must be maintained **as low as reasonably achievable**. Therefore, unless they are known to be non-hazardous, all samples should be opened, transferred and prepared in a fume hood, or under other means of mechanical ventilation. 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 will 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. Aeration Tubing: A straight glass frit having a course porosity and Tygon tubing is used for the transfer of mercury vapor from the sample bottle to the absorption cell and return.
 - 6.2.7. Drying device (a drying tube containing magnesium perchlorate or magnesium sulfate and/or a lamp with a 60 W bulb) to prevent condensation in cell. The lamp is positioned to shine on the absorption cell maintaining the air temperature in the cell about 10 °C above room temperature. Other drying devices that acheive the same purpose are also acceptable (i.e., Gortex filter).
- Note:** Instruments designed specifically for the measurement of mercury using the cold vapor technique may be substituted for the atomic absorption spectrophotometer.
- 6.3. BOD bottles or equivalent
 - 6.4. Nitrogen or argon gas supply, welding grade or equivalent
 - 6.5. Calibrated automatic pipettes or Class A glass volumetric 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 (in 10% HNO₃) 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.
- 7.3. Working mercury standard (0.1 ppm): Take 1 mL of the stock mercury standard (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% HNO₃. This acid (150 uL of concentrated HNO₃) must be added to the flask/bottle before the addition of the stock standard aliquot.
- 7.4. The calibration standards must be prepared fresh daily from the working standard (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

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 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 (HNO₃), concentrated, trace metal grade or better.

Note: If a high reagent blank is obtained, it may be necessary to distill the nitric acid.

- 7.8. Sulfuric acid (H₂SO₄), concentrated, trace metal grade or better.

- 7.9. Hydrochloric acid (HCl), concentrated, trace 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 HNO₃.
- 7.11. Stannous chloride solution: Add 50 g 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 12 g of sodium chloride and 12 g of hydroxylamine hydrochloride to every 100 mL of reagent water.

Note: Hydroxylamine sulfate may be used in place of hydroxylamine hydrochloride.

- 7.13. Potassium permanganate, 5% solution (w/v): Dissolve 5 g of potassium permanganate for every 100 mL of reagent water.

8. **SAMPLE COLLECTION, PRESERVATION AND STORAGE**

- 8.1. Sample holding time for mercury is 28 days from time of collection to the time of sample analysis.
- 8.2. Soil samples do not require preservation but must be stored at 4° C ± 2° C 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

Prior to the analysis of any analyte using 7471A, 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 MDL is determined using seven replicates of reagent water, spiked with all the analytes of interest, that have been carried through the entire analytical procedure. MDLs must be redetermined on an annual basis in accordance with 40 CFR Part 136 Appendix B requirements. The result of the MDL determination must be below the TestAmerica North Canton reporting limit.

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. The results of the initial demonstration study must be acceptable before analysis of samples may begin.

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.

- 9.2. 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, a 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 included in the sample count for determining the size of a preparation batch.
- 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. The method blank should not contain any analyte of interest at or above the reporting 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). Note: For Ohio VAP projects, the result must be below the reporting limit or samples must be redigested.
- Repreparation and reanalysis 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 reanalysis 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 aqueous LCS must be processed with each preparation batch. The LCS is used to monitor the accuracy of the analytical process. On-going 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. Until in-house control limits are established, a control limit of 70-130% recovery must be applied.

- 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.
- 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 the matrix spike RPD limits.
- Corrective action will be reparation and reanalysis of the batch unless the client agrees that other corrective action is acceptable.

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 (DQO's) may require the use of sample duplicates in place of or in addition to MS/MSD's. 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 A).

- 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. 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 reparation and reanalysis 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 4 times the spike level for that analyte, the recovery data are reported as NC (i.e., not calculated). If the reporting software does not have the ability to report NC then the actual recovery must be reported and narrated as follows: "Results outside of limits do not necessarily reflect poor method performance in the matrix due to high analyte concentrations in the sample relative to the spike level."

- If an MS/MSD is not possible due to limited sample volume, then a laboratory control sample duplicate (LCSD) should be analyzed. The RPD of the LCS and LCSD must be compared to the matrix spike RPD limits.
- 9.7. 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. If either the ICV or ICB fail to meet criteria, the analysis should be terminated, the problem corrected and the instrument recalibrated. (See Section 11.2.10 and Section 11.2.11 for required run sequence). If the cause of the ICV or ICB failure was not directly instrument related the corrective action will include reparation of the ICV, ICB, CRA, CCV and CCB with the calibration curve.
- 9.8. 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. The CCV result must fall within 20% of the true value for that solution. A CCB is analyzed immediately following each CCV. (See Section 11.2.10 and 11.2.11 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 ICV/CCV and ICB/CCB pairs.
- 9.9. 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. Recovery must be $\pm 50\%$ 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. (See Section 11.2.8 for the required run sequence.)
- 9.10. Method of Standard Addition (MSA) -This technique involves adding known amounts of standard to one or more aliquots of the sample prior to preparation. This technique compensates for a sample interferent that may enhance or depress the analyte signal, thus producing a different slope from that of the calibration standards. It will not correct for additive interferences which cause a baseline shift. Refer to Section 11.2.12 for additional information on when full 4 point MSA is required as well as Appendix C for specific MSA requirements.
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 the TestAmerica North Canton reporting limit. Analyze standards in ascending order beginning with the blank. Refer to Section 7.4 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 shall be stopped and recalibrated prior to running samples. Sample results can not be reported from a curve with an unacceptable correlation coefficient. NOTE: If any digested standard does not meet SW846 criteria, all associated Ohio VAP samples must be redigested.
- 10.7. Refer to Section 9.0 for calibration verification procedures, acceptance criteria and corrective actions.

11. **PROCEDURE**

11.1. Standard and Sample Preparation:

- 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 (7.3) into a series of sample digestion bottles. 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.

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 11.1.5 below.

11.1.4. Transfer 0.6 g of a well mixed sample into a clean sample digestion bottle. Continue preparation as described under Section 11.1.5.

11.1.5. Water Bath protocol

11.1.5.1. To each **standard** bottle: Add 5 mL of aqua regia.

11.1.5.2. To each **sample** bottle: Add 10 mL of reagent water and 5 mL of aqua regia.

11.1.5.3. Heat for two minutes in a water bath at 90 - 95 ° C.

11.1.5.4. Add 40 mL of distilled water.

11.1.5.5. Add 15 mL of potassium permanganate solution.

11.1.5.6. Heat for 30 minutes in the water bath at 90 - 95 °C.

11.1.5.7. Cool.

11.1.5.8. Add 6 mL of sodium chloride-hydroxylamine sulfate solution to reduce the excess permanganate.

11.1.5.9. To each **standard** bottle: Add 50 mL of reagent water.
To each **sample** bottle: Add 50 mL of reagent water.

11.1.5.10. Continue as described under Section 11.2.

11.2. Sample Analysis

11.2.1. Because of differences between various makes and models of CVAA instrumentation, no detailed operating instructions can be provided. Refer to the facility specific instrument operating SOP and the CVAA instrument manual for detailed setup and operation protocols.

11.2.2. All labs are required to detail the conditions/programs utilized for each instrument within the facility specific instrument operation SOP.

11.2.3. Manual determination:

11.2.3.1. Treating each sample individually, purge the headspace of the sample bottle for at least one minute.

- 11.2.3.2. Add 5 mL of stannous chloride solution and immediately attach the bottle to the aeration apparatus.
- 11.2.3.3. Allow the sample to stand quietly without manual agitation while the sample is aerated (1 L/min flow). Monitor the sample absorbance during aeration. When the absorbance reaches a maximum and the signal levels off, open the bypass valve and continue aeration until the absorbance returns to its baseline level. Close the bypass valve and remove the aeration device.
- 11.2.3.4. Place the aeration device into 100 mLs of 1% HNO₃ and allow to bubble rinse until the next sample is analyzed.
- 11.2.4. Automated determination: Refer to Appendix G for instrument setup and operation.
- 11.2.5. Perform a linear regression analysis of the calibration standards by plotting maximum response of the standards vs. micrograms (ug) of mercury. Determine the mercury concentration in the samples from the linear regression fit of the calibration curve. Calibration using computer or calculation based regression curve fitting techniques on concentration/response data is acceptable.
- 11.2.6. All measurements must fall within the defined calibration range to be valid. Dilute and reanalyze all samples for analytes that exceed the highest calibration standard.
- 11.2.7. If the sample results are negative and the absolute value of the negative result is greater than the reporting limit, the sample must be diluted and reanalyzed.
- 11.2.8. The samples must be allowed to cool to room temperature prior to analysis or a decrease in the response signal can occur.
- 11.2.9. Baseline correction is acceptable as long as it is performed after every sample or after the CCV and CCB; resloping is acceptable as long as it is immediately preceded and followed by a compliant CCV and CCB.
- 11.2.10. The following analytical sequence must be used with Method 7471A:

Instrument Calibration

ICV

ICB

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 to apply to Method 7471A.

Note: Samples include the method blank, LCS, MS, MSD, duplicate, field samples and sample dilutions.

- 11.2.11. The following run sequence is consistent with Method 7471A and may be used as an alternate to the sequence in 11.2.10. This run sequence is recommended if multiple method requirements must be accommodated in one analytical run:

Instrument Calibration

ICV

ICB

CRA

CCV

CCB

10 samples

CCV

CCB

Repeat sequence of 10 samples between CCV/CCB pairs as required to complete run.

CCV

CCB

- 11.2.12. For TCLP samples, full four point MSA will be required if all of the following conditions are met:

- 1) Recovery of the analyte in the matrix spike is not at least 50%,
- 2) The concentration of the analyte does not exceed the regulatory level, and,
- 3) The concentration of the analyte is within 20% of the regulatory level.

Appendix E provides guidance on performing MSA analyses. For TCLP mercury determinations, MSA spikes must be added prior to sample preparation.

- 11.3. 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.

- 11.4. Guidelines are provided in the appendices on procedures to minimize contamination of

samples and standards, preventive maintenance and troubleshooting.

- 11.5. One time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using a Nonconformance Memo. The Nonconformance Memo shall be filed in the project file.
- 11.6. Any unauthorized deviations from this procedure must also be documented as a nonconformance, with a cause and corrective action described.

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. For automated determinations, 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 should be omitted from the above equation.

- 12.6. For manual (total) determinations, the final concentration determined in solid samples when reported on a dry weight basis is calculated as follows:

$$mg/kg, dry weight = (C) / (W \times S)$$

Where:

C = Concentration (ug) from instrument readout

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 should be omitted from the above equation.

- 12.7. The LCS percent recovery is calculated according to the following equation:

$$\%R = 100 \left(\frac{Found(LCS)}{True(LCS)} \right)$$

- 12.8. Sample results should be reported with up to three significant figures in accordance with the TestAmerica North Canton significant figure policy.

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. Method performance is determined by the analysis of method blank, laboratory control sample, matrix spike and matrix spike duplicate samples. The matrix spike recovery should fall within +/- 30 % and the matrix spike duplicates should compare within 20% RPD. The method blanks must meet the criteria in Section 9.4. The laboratory control sample should recover within 20% of the true value until in house limits are established.
- 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. This method does not contain any specific modifications that serve to minimize or prevent pollution.

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 Safety Manual 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".

16. REFERENCES

16.1. References

- 16.1.1. Test Methods for Evaluating Solid Waste , Physical/Chemical Methods, SW-846, 3rd Edition, Final Update II, Revision I, September 1994, Method 7471A (Mercury).
- 16.1.2. Corporate Quality Management Plan (QMP), current version.
- 16.1.3. TestAmerica Laboratory Quality Manual (LQM), current version. Associated SOPs and Policies, latest version
- 16.1.4. TestAmerica Corporate Safety Manual, M-E-0001 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-0014
- 16.2.3. Statistical Evaluation of Data and Development of Control Charts, NC-QA-0018
- 16.2.4. Method Detection Limits and Instrument Detection Limits, NC-QA-0021 and S-Q-003.
- 16.2.5. Supplemental Practice for DoD Project Work, SOP NC-QA-0016
- 16.2.6. Standards and Reagents, SOP NC-QA-0017.

17. MISCELLANEOUS (TABLES, APPENDICES, ETC.)

17.1. Modifications/Interpretations from reference method.

17.1.1. Modifications from Method 7471A

- 17.1.1.1. A potassium persulfate oxidation step has been included to facilitate the breakdown of organic mercurials which are not completely oxidized by potassium permanganate. Use of potassium persulfate in combination

with the permanganate improves the recovery of mercury from organo-mercury compounds. The use of persulfate has been incorporated in several recent EPA mercury protocols.

- 17.1.1.2. The alternate run sequence presented in Section 11.2.11 is consistent with method requirements. An additional QC analysis (CRA) was added to accommodate the CLP protocol requirements.

17.1.2. Modifications from Method 7471A

- 17.1.2.1. 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.
- 17.1.2.2. Chapter 1 of SW-846 states that the method blank should 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.

17.2. Documentation and Record Management

The following documentation comprises a complete CVAA raw data package:

- Raw data (direct instrument printout)
- Run log printout from instrument software where this option is available or manually generated run log. (A bench sheet may be substituted for the run log as long as it contains an accurate representation of the analytical sequence).
- Data review checklist - See Appendix B
- Standards Documentation (source, lot, date).
- Copy of digestion log.
- Nonconformance summary (if applicable).

Figure 1. Solid Sample Preparation for Mercury - Water Bath Procedure

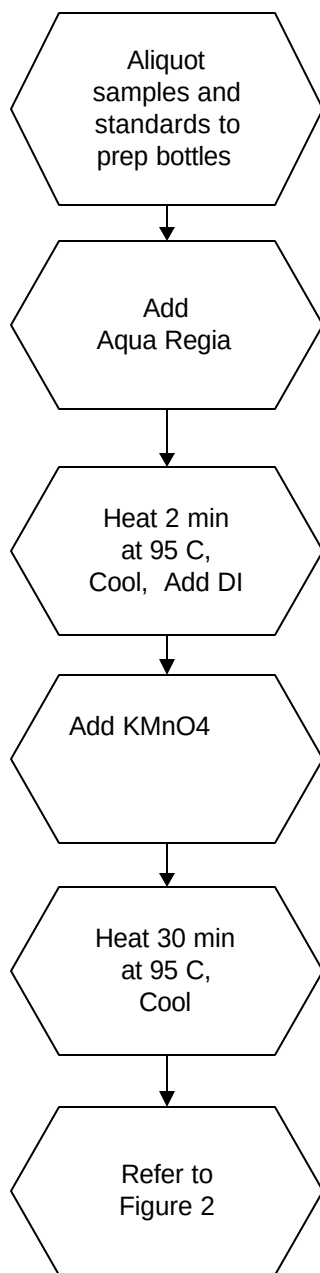
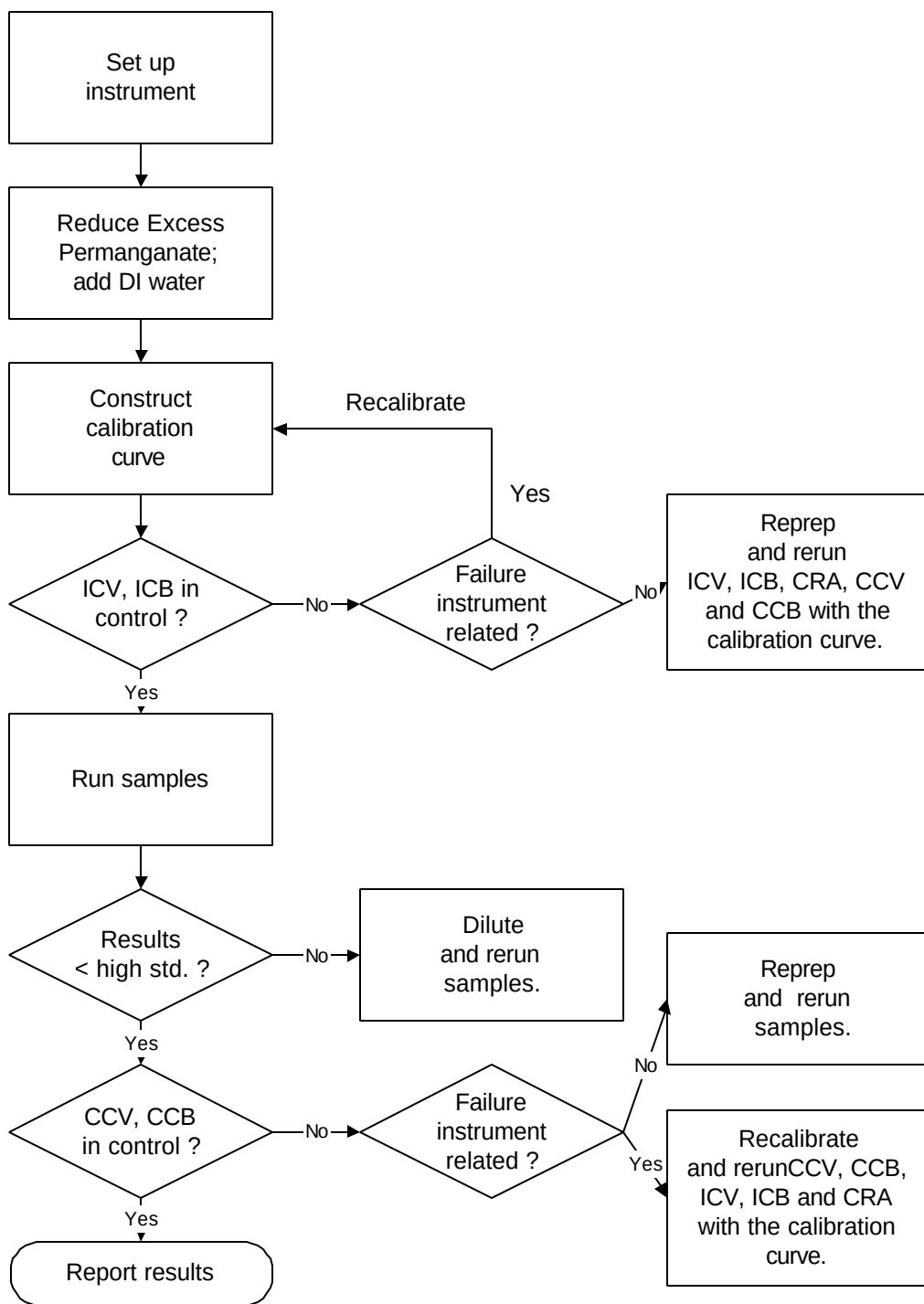


Figure 2. CVAA Mercury Analysis



APPENDIX A

TABLES

TABLE I. MERCURY REPORTING LIMITS, CALIBRATION STANDARD*, QC STANDARD AND SPIKING LEVELS

Soil RL (mg/kg)	0.1
Std 0 (mg/L)	0
Std 1 (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)	0.001 or 0.0025 ***
CCV/LCS/LCSD (mg/L)	0.0025 or 0.005 ***
MS (mg/L)	0.001

- * SOP specified calibration levels must be used unless prevented by the instrument configuration or client specific requirements. Deviations from specified calibration levels must be documented in the facility specific instrument operation SOP and must be approved by the facility technical manager and Quality Assurance Manager.
- ** Optional standard which may be used to extend the calibration range as allowed by the instrument configuration. If the instrument configuration prevents the use of 6 standards, the 2 ppb standard may be eliminated in favor of the 10 ppb standard.
- *** Concentration level dependent on high calibration standard used. CCV must be 50% of the high standard concentration and the ICV must be 20-25% of the high standard concentration.

TABLE II. Summary Of Quality Control Requirements

QC Parameter	Frequency *	Acceptance Criteria	Corrective Action
ICV	Beginning of every analytical run	90 - 110 % recovery	Terminate analysis; Correct the problem; Recalibrate or reprep with calibration curve (see Section 9.7)
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 9.7)
CCV	Every 10 samples and at the end of the run	80 - 120 % recovery	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 Sections 9.8 and 10.6).
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. If CCB is biased high and samples are ND, results can be reported (see Section 9.8).
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

*See Sections 11.2.10 and 11.2.11 for exact run sequence to be followed.

TABLE II. Summary of Quality Control Requirements (Cont'd)

QC Parameter	Frequency *	Acceptance Criteria	Corrective Action
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	Terminate analysis, correct the problem, redigest and reanalyze 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 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 > 4x the spike added. (see Section 9.6) For TCLP see Section 11.3.12
Matrix Spike Duplicate	See Matrix Spike	75 - 125 % recovery or in-house control limits RPD $\leq$ 20% (see MS)	See Corrective Action for Matrix Spike

APPENDIX B

EXAMPLE

TESTAMERICA NORTH CANTON Hg DATA REVIEW CHECKLIST

Example
TestAmerica North Canton Hg Data Review Checklist

Run/Project Information

Run Date: _____ Analyst: _____ Instrument: _____
Prep Batches Run: _____

Circle Methods used: 7470A / 245.1 : CORP-MT-0005 Rev 1 7471 : CORP-MT-0007 Rev 1

Review Items

A. Calibration/Instrument Run QC	Yes	No	N/A	2ndLevel
1. Instrument calibrated per manufacturer's instructions and at SOP specified levels ?				
2. ICV/CCV analyzed at appropriate frequency and within control limits?				
3. ICB/CCB analyzed at appropriate frequency and within +/- RL or +/- CRDL (CLP)?				
4. CRA run (CLP only)?				
B. Sample Results				
1. Were samples with concentrations > the high calibration standard diluted and reanalyzed?				
2. All reported results bracketed by in control QC ?				
3. Sample analyses done within holding time?				
C. Preparation/Matrix QC				
1. LCS done per prep batch and within QC limits ?				
2. Method blank done per prep batch and < RL or CRDL (CLP) ?				
3. MS run at required frequency and within limits ?				
4. MSD or DU run at required frequency and RPD within SOP limits?				
D. Other				
1. Are all nonconformances documented appropriately ?				
2. Current IDL/MDL data on file?				
3. Calculations and Transcriptions checked for error ?				
4. All client/ project specific requirements met?				
5. Date of analysis verified as correct ?				

Analyst: _____ Date: _____
Comments:

2nd Level Reviewer : _____ Date: _____

APPENDIX C
MSA GUIDANCE

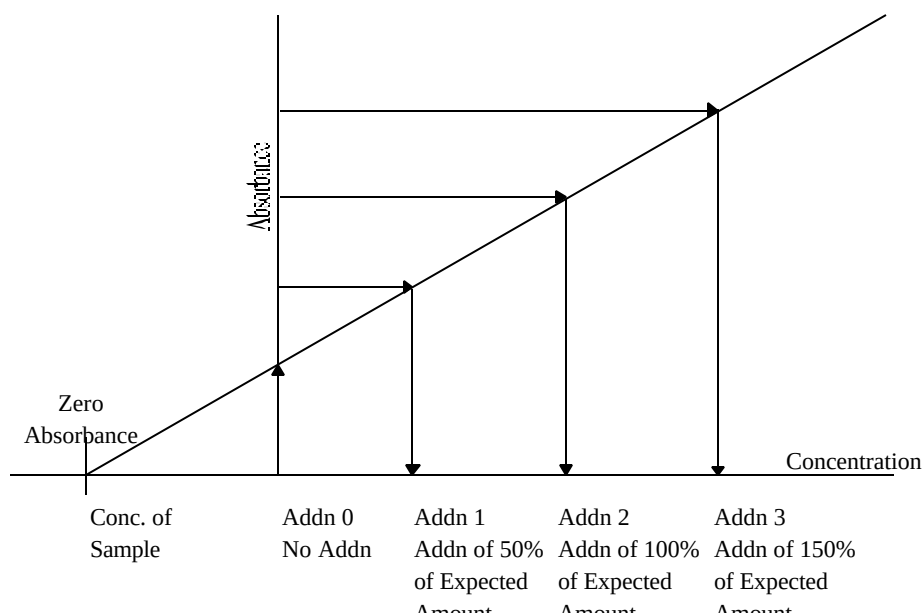
APPENDIX C. MSA GUIDANCE

Method of Standard Addition

Four equal volume aliquots of sample are measured and known amounts of standards are added to three aliquots. The fourth aliquot is the unknown and no standard is added to it. The concentration of standard added to the first aliquot should be 50% of the expected concentration. The concentration of standard added to the second aliquot should be 100% of the expected concentration and the concentration of standard added to the third aliquot should be 150% of the expected concentration. The volume of the unspiked and spiked aliquots should be the same (i.e., the volume of the spike added should be negligible in relation to the volume of sample).

To determine the concentration of analyte in the sample, the absorbance (or response) of each solution is determined and a linear regression performed. On the vertical axis the absorbance (or response) is plotted versus the concentrations of the standards on the horizontal axis using 0 as the concentration of the unspiked aliquot. An example plot is shown in Figure 1. When the resulting line is extrapolated back to zero absorbance, the point of interception of the horizontal axis is the concentration of the unknown. Calculate the correlation coefficient (r) and the x-intercept (where $y=0$) of the curve. The concentration in the digestate is equal to the negative x-intercept.

Figure 1



- For the method of standard additions to be correctly applied, the following limitations must be taken into consideration.
- The plot of the sample and standards must be linear over the concentration range of concern. For best results, the slope of the curve should be similar to that of a plot of the aqueous standard curve.
- The effect of the interference should not vary as the ratio of the standard added to the sample matrix changes.

APPENDIX D
TROUBLESHOOTING GUIDE

APPENDIX D. TROUBLESHOOTING GUIDE

Problem	Possible Cause
Poor or No Absorbance or Sensitivity Check failed	Incorrect wavelength Dirty windows Window loose Etched or dirty optics Wrong lamp Bad lamp Not enough or no sample introduced Empty sample cup Incorrectly made standards Gas leak EDL power supply set on "Continuous"
Erratic Readings	Source lamp not aligned properly Lamp not prewarmed Injection tip partially clogged Contaminated reagents Contaminated glassware Drying tube saturated Bad lamp Injection tip hitting outside of tube Injection tip coated or not set properly Leak in sample tubing Power fluctuations Air bubbles in tubing
EDL Won't Light	Lamp cable not plugged in Lamp power set at 0 Lamp is dead Power supply fuse is blown Short in cord
Standards reading twice or half normal absorbance or concentration	Incorrect standard used Incorrect dilution performed Dirty cell
Background Correction Light Blinking	Background screen or attenuator faulty

APPENDIX E
CONTAMINATION CONTROL GUIDELINES

APPENDIX E. CONTAMINATION CONTROL GUIDELINES

The following procedures are strongly recommended to prevent contamination:

All work areas used to prepare standards and spikes should be cleaned before and after each use.

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 must be kept scrupulously clean.

Powdered or Latex Gloves must not be used in the metals laboratory since the powder contains silica and zinc, as well as other metallic analytes. Only vinyl or nitrile gloves should be used in the metals laboratory.

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.

APPENDIX F
PREVENTIVE MAINTENANCE

APPENDIX F. PREVENTIVE 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.

Cold Vapor Atomic Absorption (Leeman PS 200) ¹

Daily	Semi-annually	Annually
Clean lens	Check Hg lamp intensity	Change Hg lamp
Check aperture		Check liquid/gas separator
Check argon flow		
Check tubing		
Check drain		
Replace drying tube		

Cold Vapor Atomic Absorption (PE 5000) ¹

Daily	Monthly
Clean aspirator by flushing with DI water	Clean cell in aqua regia
Check tubing and replace if needed	Clean aspirator in aqua regia
Clean windows with methanol	
Change silica gel in drying tube	
Check argon gas supply	
Adjust lamp	

APPENDIX G
INSTRUMENT SET UP

Hg Analysis (Leeman PS200II)

SYSTEM INITIALIZATION AND WARM UP

1. F1 Menu
2. Instrument
 - a. Taskmaster
 - b. #4 Wake System Up - Enter

The warming up period takes approximately ten minutes.

TO SET UP INSTRUMENT FOR ANALYSIS

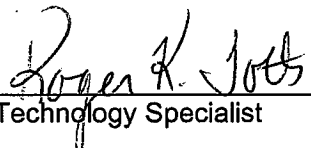
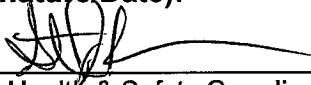
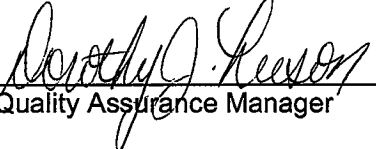
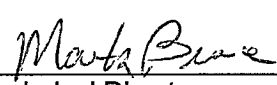
1. F1 Menu
2. Autosampler
 - A. Rack Entry
 - B. Edit (ex. Rack 1), Enter
 - C. Cup ID - Enter (clears sample #'s)
 - D. Extended ID- type in matrix of sample (water or solid), Enter.
 - E. Press Insert Key and move cursor with arrows to cup ID and begin typing labels.
 - F. F3 Print Screen
3. Press F2 Macro key and type in analyst's first name - Enter
 - 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.

**Title: INDUCTIVELY COUPLED PLASMA – ATOMIC EMISSION
SPECTROSCOPY, SPECTROMETRIC METHOD FOR TRACE ELEMENT
ANALYSES**

[SW846 Method 6010B and EPA Method 200.7]

Approvals (Signature/Date):			
	1-14-08		1-14-08
Technology Specialist	Date	Health & Safety Coordinator	Date
	1/14/08		1/14/08
Quality Assurance Manager	Date	Laboratory Director	Date
	1/14/08		
Technical Director	Date		

This SOP was previously identified as SOP CORP-MT-0001NC, Rev 3.4, dated 01/08/04

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TABLE OF CONTENTS

1. SCOPE AND APPLICATION.....	3
2. SUMMARY OF METHOD.....	3
3. DEFINITIONS	4
4. INTERFERENCES.....	4
5. SAFETY.....	5
6. EQUIPMENT AND SUPPLIES	6
7. REAGENTS AND STANDARDS.....	7
8. SAMPLE COLLECTION, PRESERVATION AND STORAGE	7
9. QUALITY CONTROL.....	8
10. CALIBRATION AND STANDARDIZATION	14
11. PROCEDURE	15
12. DATA ANALYSIS AND CALCULATIONS	19
13. METHOD PERFORMANCE	22
14. POLLUTION PREVENTION	22
15. WASTE MANAGEMENT	22
16. REFERENCES.....	22
17. MISCELLANEOUS (TABLES, APPENDICES, ETC.).....	23

LIST OF APPENDICES:

APPENDIX A - TABLES	29
APPENDIX B - CROSS REFERENCE OF TERMS USED IN METHODS AND IN SOP.....	38
APPENDIX C - MSA GUIDANCE	40
APPENDIX D - TROUBLESHOOTING GUIDE	42
APPENDIX E - CONTAMINATION CONTROL GUIDELINES	44
APPENDIX F - PREVENTATIVE MAINTENANCE.....	46
APPENDIX G – ICP OPERATING INSTRUCTIONS.....	48

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 Method 6010B and EPA Method 200.7. Table I of Appendix A lists the elements appropriate for analysis by Methods 6010B and 200.7. Additional elements may be analyzed under Methods 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. Method 6010B is 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 with the exception of analyses for dissolved metals in filtered and acidified aqueous samples. Although digestion is not specifically required by the method, some clients and regulators may require digestion of **dissolved samples** and this must be clarified and documented before project initiation. 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 with the exception of analyses for dissolved metals in filtered and acidified aqueous samples if the criteria in Section 11.1 are met. 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 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 should 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.

- 2.2. Refer to CORP-IP-0002NC, Acid Digestion of Soils, SW846 Method 3050B and CORP-IP-0003NC, 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. Suspended Metals: Those elements which are retained by a 0.45 um membrane.
- 3.3. Total Metals: The concentration determined on an unfiltered sample following vigorous digestion.
- 3.4. Total Recoverable Metals: The concentration determined on an unfiltered sample following treatment with hot, dilute mineral acid.

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.
- 4.1.4. 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 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 will be removed and discarded; other gloves will 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 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

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 should not go near the instrument while in operation.

5.4. Exposure to chemicals must be maintained **as low as reasonably achievable**; therefore, unless they are known to be non-hazardous, all samples should be opened, transferred and prepared in a fume hood, or under other means of mechanical ventilation, where possible. 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 will 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 μ L $\rightarrow$ 10 ml
- 6.7. Class A volumetric flasks – range from 50 ml $\rightarrow$ 2000 ml
- 6.8. Autosampler tubes – range from 8 ml $\rightarrow$ 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.
- 7.2. Working calibration and calibration verification solutions may be used for up to 3 months and must be replaced sooner if verification from an independent source indicates a problem. Standards should be prepared in a matrix of 5% hydrochloric and 5% nitric acids. Refer to Tables III, IV, IVA, V and VI (Appendix A) for details regarding the working standard concentrations for calibration, calibration verification, interference correction and spiking solutions.
- 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 or silica are 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 $4^{\circ}\text{C} \pm 2^{\circ}$ until the time of preparation.

9. QUALITY CONTROL

Table VII (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 either Method 200.7 or Method 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. If the instrument is adjusted in anyway that may affect the IDL, the IDL for that instrument must be redetermined. The IDL shall 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). The result of the IDL determination must be below the TestAmerica North Canton reporting limit.
- 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-0021 and S-Q-003 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 should 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.

- 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 interelement 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 interelement 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 should 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 $\pm$ the RL as defined in Tables I, IA or II. For elements with a reporting limit of 10 ug/L or less, the signal should be $\pm$ 2X 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 9.1.4) should 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 reanalyzed 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 should 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 contamination level). For Ohio VAP projects, all analytes must be less than the reporting limit.

- 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 reanalysis 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 reanalysis is not possible, then the sample data must be qualified. This anomaly must be addressed in the project narrative.
- For dissolved metal samples, which have not been digested, a CCB result is

reported as the method blank. The CCB run immediately prior to the start of the dissolved sample analyses must be used for this purpose. No more than 20 samples can be associated with one CCB.

- 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 III (Appendix A). The LCS is used to monitor the accuracy of the analytical process. On-going 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 the matrix spike RPD 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 reanalysis of the batch unless the client agrees that other corrective action is acceptable.
 - For dissolved metals samples which have not been digested, a CCV result is reported as the LCS. The CCV run immediately prior to the start of the dissolved sample analyses must be used for this purpose. No more than 20 samples can be associated with one CCV.
- 9.4. 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 III and VI (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 both methods 200.7 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 reparation and reanalysis 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 4x the spike level for that analyte, the recovery data is reported as NC, MSB (i.e., not 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) should be analyzed. The RPD of the LCS and LCSD must be compared to the matrix spike RPD limits.
- For dissolved metals samples which have not been digested, a MS/MSD must be performed per batch of up to 20 samples by spiking two aliquots of the sample at the levels specified in Table III (Appendix A).

- 9.5. 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 5x (1:4) dilution. Samples identified as field blanks cannot be used for dilution tests. The results of the diluted sample, after correction for dilution, should agree within 10% of the original sample determination when the original sample concentration is greater than 50x the IDL. If the results are not within 10%, the possibility of chemical or physical interference exists and the data is flagged.
- 9.6. 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 with relative standard deviation <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 should be terminated, the problem corrected, the instrument recalibrated and the calibration reverified. (See Section 11.8 or 11.11

for required run sequence).

- 9.7. Continuing 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 a mid-range standard made from a dilution of the calibration standard. The CCV for both methods must fall within 10% of the true value for that solution with relative standard deviation <5% from replicate (minimum of two) exposures. A CCB is analyzed immediately following each CCV. (See Section 11.8 or 11.11 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 11.13 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.
- 9.8. Interference Check Analysis (ICSA/ICSAB) - The validity of the interelement 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 V (Appendix A) for the details of ICSA and ICSAB composition. Custom multielement ICS solutions must be used. All analytes should be spiked into the ICSAB solution, therefore, if a non-routine analyte is required then it should 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 9.8.3. 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.
- 9.8.1. The ICSA and ICSAB solutions must be run at the beginning of the run. (See Section 11.10 or 11.13 for required run sequence.)
- 9.8.2. The ICSAB results for the interferents must fall within 80 - 120% of the true value. If any ICSAB interferent result fails criteria, the analysis should be terminated, the problem corrected, the instrument recalibrated and the samples rerun.
- 9.8.3. ICSA results for the non-interfering elements with reporting limits ≤ 10 ug/L must fall within the TestAmerica North Canton guidelines of $\pm 2x$ RL from zero. ICSA results for the non-interfering elements with RLs > 10 μ g/L must fall within the TestAmerica North Canton guidelines of $\pm 1x$ RL from zero. If the ICSA results for the non-interfering elements do not fall within +/- 2x RL (RL ≤ 10) or $\pm 1x$ RL (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 $\pm 2x$ 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 $\pm 2x$ RL from zero, the data can be accepted only if the concentration of the affected analyte in the field sample is more than 10x 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 reanalyzed 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.

- 9.9. 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 Section 11.10 or 11.13 for required run sequence.) Evaluate associated samples based upon advisory limits of $\pm 50\%$ of true value.

Note: The custom CRI mix contains most analytes at a level near the standard lab reporting limit.

- 9.10. Method of Standard Addition (MSA) -This technique involves adding known amounts of standard to one or more aliquots of the processed sample solution. This technique compensates for a sample interferent that may enhance or depress the analyte signal, thus producing a different slope from that of the calibration standards. It will not correct for additive interferences which cause a baseline shift. Refer to Section 11.17 for additional information on when MSA is required as well as Appendix C for specific MSA requirements.

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 G.
- 10.2. 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 G for detailed set up and operation protocols. Refer to Instruction Manuals in laboratory.

- 10.3. 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. Refer to Section 9.0 for calibration verification procedures, acceptance criteria and corresponding corrective actions.

11. PROCEDURE

- 11.1. For 200.7 analyses, dissolved (preserved) samples must be digested unless it can be documented that the sample meets all of the following criteria:
 - A. Visibly transparent with a turbidity measurement of 1 NTU or less.
 - B. Is of one liquid phase and free of particulate or suspended matter following acidification.
 - C. Is NOT being analyzed for silver.
- 11.2. 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.
- 11.3. Prior to calibration and between each sample/standard the system is rinsed with the calibration blank solution. The minimum rinse time between analytical samples is 60 seconds unless following the protocol outlined in 9.1.5 it can be demonstrated that a shorter rinse time may be used.
- 11.4. 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.
- 11.5. 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.
- 11.6. To facilitate the data review and reporting processes it is strongly recommended that all necessary dilutions be performed before closing out the instrument run.
- 11.7. The use of an internal standard is **required** on the Trace ICP unless the calibration and QC standards are matrix matched to each digestion procedure used as follows:

Preparation Method	% HNO ₃	% HCl
SW846 3050	10	10
SW846 3005	2	5
SW846 3010	6	5

The following procedural guidelines must be followed when using an internal standard:

- 11.7.1. Typically used internal standards are: yttrium. (Note: Any element can be used that is not typically found in environmental samples at a high rate of occurrence.)
- 11.7.2. 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.
- 11.7.3. 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.
- 11.7.4. The internal standard raw intensity counts must be printed on the raw data.
- 11.7.5. 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.
 - 11.7.5.1. If the internal standard counts fall within $\pm 30\%$ of the counts observed in the ICB then the data is acceptable.
 - 11.7.5.2. If the internal standard counts in the field samples are more than $\pm 30\%$ higher than the expected level, the field samples must then be diluted and reanalyzed.

- 11.8. 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 VII (Appendix A) for Methods 6010B
and 200.7 quality control criteria.

- 11.9. Full method required QC must be available for each wavelength used in determining
reported analyte results.
- 11.10. 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 9.7.

Original Run: Instrument Calibration

ICV

ICB

CRI

ICSA

ICSAB

CCV

CCB

10 samples

CCV

CCB

10 samples

CCV

CCB

10 samples **

CCV *

CCB *

* Failure occurs at CCV/CCB

**Samples requiring rerun for affected analytes

10 samples **
CCV
CCB
10 samples
CCV
CCB

- 11.11 The instrument may be reprofiled between CCV/CCB pairs to correct for environment induced drift.
- 11.12 Guidelines are provided in the appendices on procedures to minimize contamination of samples and standards, preventive maintenance and troubleshooting.
- 11.13 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 or use an alternate wavelength for which QC data are established. 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 a interferent for any requested analyte in that sample, the sample must be diluted. Acid strength must be maintained in the dilution of samples.
- 11.14 For TCLP samples, full four-point MSA will be required if all of the following conditions are met:
- 1) Recovery of the analyte in the matrix spike is not at least 50%,
 - 2) The concentration of the analyte does not exceed the regulatory level, and,
 - 3) The concentration of the analyte is within 20% of the regulatory level.
- The reporting and regulatory limits for TCLP analyses as well as matrix spike levels are detailed in Table VI (Appendix A). Appendix C provides guidance on performing MSA analyses.
- 11.15 Any variation in procedure shall 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.
- 11.16 Nonconformance documentation shall be filed in the project file.
- 11.17 Any unauthorized deviations from this procedure must also be documented as a nonconformance, with a cause and corrective action described.
- 11.18 Analytical Documentation

- 11.18.1 Record all analytical information in the analytical logbook/logsheets 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.
- 11.18.2 All standards are logged into a department standard logbook. All standards are assigned an unique number for identification. Logbooks are reviewed by the supervisor or designee.
- 11.18.3 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.
- 11.18.4 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:

$$\%R = 100 \left(\frac{\text{Found(ICV)}}{\text{True(ICV)}} \right)$$

- 12.2. CCV percent recoveries are calculated according to the equation:

$$\%R = 100 \left(\frac{\text{Found(CCV)}}{\text{True(CCV)}} \right)$$

- 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 when reported on a dry weight basis is calculated as follows:

$$mg / Kg, dry weight = \frac{C \times V \times D}{W \times S}$$

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

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 wet weight basis the “S” factor should be omitted from the above equation.

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 $\times$ 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-0024.

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: $Cr^{+3} = Cr, \text{ total} - Cr^{+6}$

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, IA & II in Appendix A for the list of Method 6010B and 200.7 analytes as well as additional 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 should fall within $\pm 25\%$ and the MS/MSD should compare within 20% RPD or within the laboratory's historical acceptance limits. These criteria apply to analyte concentrations greater than or equal to 10xIDL. Method blanks must meet the criteria specified in Section 9.2. The laboratory control samples should 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. This method does not contain any specific modifications that serve to minimize or prevent pollution.

15. WASTE MANAGEMENT

- 15.1. This method does not contain any specific modifications that serve to minimize or prevent pollution.
- 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 Safety Manual for "Waste Management and Pollution Prevention."
- 15.3. Waste Streams Produced by this Method
 - 15.3.1 The following waste streams are produced when this method is carried out:
 - 15.3.2 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.
 - 15.3.3 Standards should 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 III, Revision 2, December 1996. Method 6010B.
 - 16.1.3. 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.

16.1.4. 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.

16.1.5. Corporate Quality Management Plan (QMP), current version

16.1.6. TestAmerica Laboratory Quality Manual (LQM), current version.

16.2. Associated SOPs and Policies, latest version

16.2.1. TestAmerica North Canton QC Program, QA-003

16.2.2. Glassware Washing, NC-QA-0014

16.2.3. Statistical Evaluation of Data and Development of Control Charts, NC-QA-0018

16.2.4. Method Detection Limits and Instrument Detection Limits, NC-QA-0021

16.2.5. Supplemental Practices for DoD Project Work, NC-QA-0016

16.2.6. Hexavalent Chromium (Colorimetric), NC-WC-0024

16.2.7. Acid Digestion of Soils, SW846 Method 3050B, CORP-IP-0002NC

16.2.8. Acid Digestion of Aqueous Samples by SW846 and MCAWW 200 Series Methods, CORP-IP-0003NC

16.2.10 Standards and Reagents, NC-QA-0017

17. MISCELLANEOUS (TABLES, APPENDICES, ETC.)

17.1. Modifications/Interpretations from reference method

17.1.1. Modifications/interpretations from both 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 should 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. Section 8.5 of Method 6010B and Section 9.5 of Method 200.7 recommend that 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% HNO₃/5% HCl instead of the specified 2% HNO₃/10% 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 IPC (ICSA/AB) solution immediately following calibration must verify that the instrument is within $\pm 5\%$ 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 $\pm 5\%$ 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 $\pm 10\%$ 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 200.7 indicates that the QCS (ICV) should 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 should 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 should 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 $\pm$ RL from zero for calibration blank criteria. SOP section 9.7 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 should 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 3x 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 reanalyze the previous 10 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 $\pm$ RL from zero for calibration blank criteria. See SOP Section 9.7

for a detailed description of the required corrective action procedures.

APPENDIX A

TABLES

TABLE I. Method 200.7 and 6010B Target Analyte List

Element	Symbol	CAS #	6010B Analyte	200.7 Analyte	Reporting Limit (ug/L) Water	Reporting Limit (mg/kg) Soil
Aluminum	Al	7429-90-5	X	X	200	20
Antimony	Sb	7440-36-0	X	X	60	6
Arsenic	As	7440-38-2	X	X	300	30
Barium	Ba	7440-39-3	X	X	200	20
Beryllium	Be	7440-41-7	X	X	5.0	0.5
Boron	B	7440-42-8	X	X	200	20
Cadmium	Cd	7440-43-9	X	X	5.0	0.5
Calcium	Ca	7440-70-2	X	X	5000	500
Chromium	Cr	7440-47-3	X	X	10	1
Cobalt	Co	7440-48-4	X	X	50	5
Copper	Cu	7440-50-8	X	X	25	2.5
Iron	Fe	7439-89-6	X	X	100	10
Lead	Pb	7439-92-1	X	X	100	10
Magnesium	Mg	7439-95-4	X	X	5000	500
Manganese	Mn	7439-96-5	X	X	15	1.5
Molybdenum	Mo	7439-98-7	X	X	40	4
Nickel	Ni	7440-02-0	X	X	40	4
Potassium	K	7440-09-7	X	X	5000	500
Selenium	Se	7782-49-2	X	X	250	25
Silver	Ag	7440-22-4	X	X	10	1
Sodium	Na	7440-23-5	X	X	5000	500
Thallium	Tl	7440-28-0	X	X	2000	200
Vanadium	V	7440-62-2	X	X	50	5
Zinc	Zn	7440-66-6	X	X	20	2

TABLE IA. Method 200.7 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. Non-Routine Analyte List

Element	Symbol	CAS #	Reporting Limit (ug/L) Water	Reporting Limit (mg/kg) Soil
Tin	Sn	7440-31-5	100	10
Titanium	Ti	7440-32-6	50	5

TABLE III. Matrix Spike and Aqueous Laboratory Control Sample Levels

ELEMENT	LCS Level (ug/L)	Matrix Spike Level (ug/L)
Aluminum	2000	2000
Antimony	500	500
Arsenic	2000	2000
Barium	2000	2000
Beryllium	50	50
Cadmium	50	50
Calcium	50000	50000
Chromium	200	200
Cobalt	500	500
Copper	250	250
Iron	1000	1000
Lead	500	500
Magnesium	50000	50000
Manganese	500	500
Molybdenum	1000	1000
Nickel	500	500
Potassium	50000	50000
Selenium	2000	2000
Silver	50	50
Sodium	50000	50000
Thallium	2000	2000
Vanadium	500	500
Zinc	500	500
Boron	1000	1000
Tin	2000	2000
Titanium	1000	1000

TABLE IV. 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

TABLE V. 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	-	10000**
Vanadium	-	500
Zinc	-	1000
Tin	-	1000
Boron		1000
Titanium		1000

* Non-routine elements not listed above should be spiked into the ICSAB at 1000 ug/L.

** Thallium level for Trace ICP should be at 1000 ug/L.

TABLE VI. 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

TABLE VII. 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 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%	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)	Evaluate associated samples.
ICSA	Beginning of every run	See Section 9.8.3	See Section 9.8.3
ICSAB	Immediately following each ICSA.	Results must be within 80 - 120% recovery.	See Section 9.8.2.

* See Sections 11.10 and 11.13 for exact run sequence to be followed.

TABLE VII. Summary of Quality Control Requirements (Cont'd)

QC Parameter	Frequency *	Acceptance Criteria	Corrective Action
Dilution Test	One per prep batch	For samples > 50x 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 9.2). Sample results greater than 20x the blank concentration are acceptable. Samples for which the contaminant is < RL may not require re-digestion or reanalysis (see Section 9.2).	Re-digest and reanalyze 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 require re-digestion or reanalysis (see Section 9.3)	Terminate analysis. Correct the problem. Re-digest and reanalyze all samples associated with the LCS.
Matrix Spike	One per sample preparation batch of up to 20 samples	75 - 125 % recovery or in-house control limits. For TCLP See Section 11.17.	In the absence of client specific requirements, flag the data. No flag required if sample level is > 4x the spike added. For TCLP, see Section 11.17.
Matrix Spike Duplicate	See Matrix Spike	75 - 125 % recovery; RPD ≤ 20% .	See Corrective Action for Matrix Spike.

APPENDIX B

**CROSS REFERENCE OF TERMS USED IN METHODS 6010B, 200.7,
AND TESTAMERICA NORTH CANTON**

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**CROSS REFERENCE OF TERMS COMMONLY USED IN
METHODS EPA 200.7, SW6010B, AND TESTAMERICA NORTH CANTON SOP**

EPA 200.7	SW6010B	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
MSA GUIDANCE

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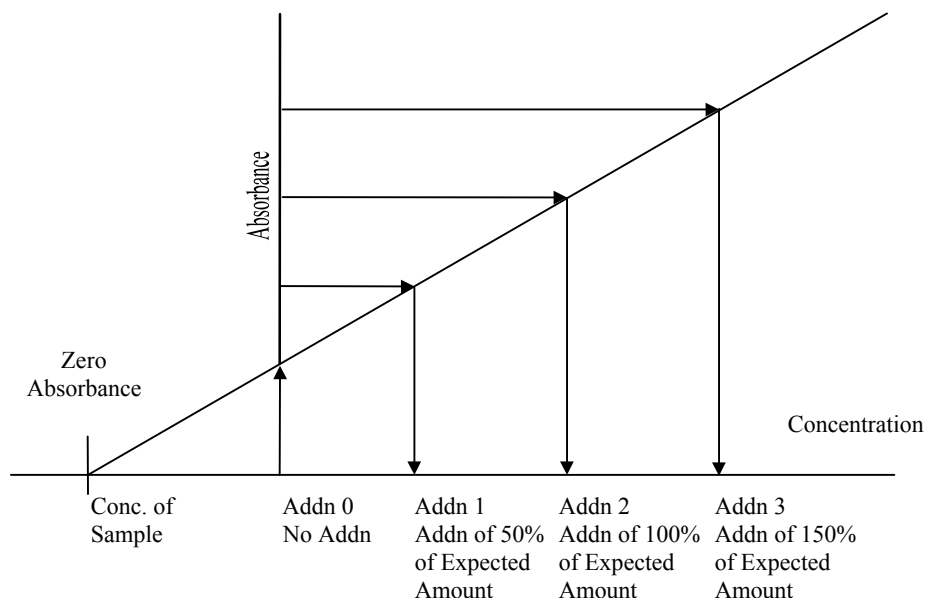
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APPENDIX C - MSA Guidance

Method of Standard Addition

Four equal volume aliquots of sample are measured and known amounts of standards are added to three aliquots. The fourth aliquot is the unknown and no standard is added to it. The concentration of standard added to the first aliquot should be 50% of the expected concentration. The concentration of standard added to the second aliquot should be 100% of the expected concentration and the concentration of standard added to the third aliquot should be 150% of the expected concentration. The volume of the unspiked and spiked standard should be the same.

In order to determine the concentration of analyte in the sample, the analytical value of each solution is determined and a plot or linear regression performed. On the vertical axis the analytical value is plotted versus the concentrations of the standards on the horizontal axis. An example plot is shown in Figure 1. When the resulting line is extrapolated back to zero absorbance, the point of interception of the horizontal axis is the concentration of the unknown.



For the method of standard additions to be correctly applied, the following limitations must be taken into consideration:

- The plot of the sample and standards must be linear over the concentration range of concern. For best results, the slope of the curve should be similar to that of a plot of the aqueous standard curve.
- The effect of the interference should not vary as the ratio of the standard added to the sample matrix changes.

APPENDIX D
TROUBLESHOOTING GUIDE

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APPENDIX D - 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 Lower Torch
Instrument Drift	RF not cooling properly Vacuum level is too low Replace torch (Crack) Clean or replace nebulizer (blockage) Check room temperature (changing) Replace pump tubing Room humidity too high Clean torch tip (salt buildup) Check for argon leaks Adjust sample carrier gas Reprofile Horizontal Mirror Replace PA tube
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

APPENDIX E
CONTAMINATION CONTROL GUIDELINES

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APPENDIX E - CONTAMINATION CONTROL GUIDELINES

The following procedures are strongly recommended to prevent contamination:

All work areas used to prepare standards and spikes should be cleaned before and after each use.

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 must be kept scrupulously clean.

Powdered or Latex Gloves must not be used in the metals laboratory since the powder contains silica and zinc as well as other metallic analytes. Only vinyl or nitrile gloves should be used in the metals laboratory.

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:

Yellow pipet tips and volumetric caps can sometimes contain cadmium.

Some sample cups have been found to contain lead.

The markings on glass beakers have been found to contain lead. If acid baths are in use for glassware cleaning, they should be periodically checked for contaminants since contaminant concentrations will increase over time.

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.

Latex gloves contain over 500 ppb of zinc.

APPENDIX F

PREVENTIVE MAINTENANCE

APPENDIX F - PREVENTIVE 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:

<u>Daily</u>	Change sample pump tubing and pump windings 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
<u>As Needed</u>	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
<u>Bi-yearly</u>	Change oil for vacuum systems Replace coolant water filter (may require more or less frequently depending on quality of cooling water)

APPENDIX G

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

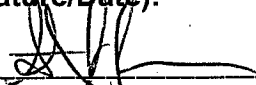
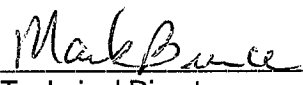
- a. Methods (enter)
- b. Enter method name
- c. (F3)-Method Info.
- d. Change file name
- e. (F9)-Done
- f. (F9)-Done/Keep

3. OPERATION

- a. Analysis (enter)
- b. (F5)-Profile
 1. (F3)-Automatic

2. (F1)-Run
 3. If peak position is greater than $\pm .05$ units from the center peak position, you must adjust the profile. If it is within $\pm .05$ 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 $\pm .05$ units.
 7. (F9)-Done
- c. Autosampler (F9)
1. Enter method name (enter)
 2. Enter autosampler table name (enter)
 3. (F1)-Run

**Title: EXTRACTION OF ORGANIC COMPOUNDS FROM WATERS
AND SOILS, BASED ON SW846 3500 SERIES, 3600 SERIES, AND 600
SERIES METHODS**

Approvals (Signature/Date):	
 Technology Specialist 3/3/08 Date	 Health & Safety Coordinator 3-6-08 Date
 Quality Assurance Manager 3/6/08 Date	 Laboratory Director 3/4/08 Date
 Technical Director 3/12/08 Date	

This SOP was previously identified as SOP No. CORP-OP-0001NC, Rev 4.2, dated 01/30/06

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TABLE OF CONTENTS

1.	SCOPE AND APPLICATION	3
2.	SUMMARY OF METHOD.....	3
3.	DEFINITIONS.....	4
4.	INTERFERENCES	4
5.	SAFETY	4
6.	EQUIPMENT AND SUPPLIES	6
7.	REAGENTS AND STANDARDS	8
8.	SAMPLE COLLECTION PRESERVATION AND STORAGE.....	9
9.	QUALITY CONTROL.....	9
10.	CALIBRATION AND STANDARDIZATION.....	9
11.	PROCEDURE.....	11
12.	DATA ANALYSIS AND CALCULATIONS	20
13.	METHOD PERFORMANCE.....	21
14.	POLLUTION PREVENTION.....	21
15.	WASTE MANAGEMENT	21
16.	REFERENCES.....	22
17.	MISCELLANEOUS.....	23

List of Tables

Table 1	Liquid / Liquid Extraction Conditions
Table 2	Exchange Solvents and Final Volumes
Table 3	Surrogate Spiking Solutions
Table 4	Matrix Spike and LCS Solutions
Table 5	Surrogate Spike Components
Table 6	Matrix Spike Components

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 SW-846 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.

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 Safety Manual, Lab Specific Addendum to the CSM, 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 will be removed and discarded, other gloves will 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'dichlorobenzidine, 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 should be prepared in 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 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
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.
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**; therefore, unless they are known to be non-hazardous, all samples should be opened, transferred and prepared in a fume hood, or under other means of mechanical ventilation, where possible. 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 will 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 will 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 should 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 should be cleaned per Glassware Washing, SOP NC-QA-0014.
- 6.2.1. 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		
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 ($\pm 5^{\circ}\text{C}$) 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, and 10 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 Pipets, 5 $\frac{3}{4}$ in, and 9in.	Ö	Ö	Ö	Ö	Ö
Aluminum foil	Ö	Ö	Ö	Ö	Ö
Paper Towels	Ö	Ö	Ö	Ö	Ö

7. 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 ₂ SO ₄), Concentrated: Reagent Grade	Ö	Ö			
Sulfuric acid (1:1): Carefully add 500 mL of H ₂ SO ₄ to 500 mL of reagent water. Mix well.	Ö	Ö			
Hydrochloric Acid (HCl)			Ö		
Organic free reagent water.	Ö	Ö			
Sodium sulfate (Na ₂ SO ₄), Granular, Anhydrous: Purify by heating at 400°C a minimum of two hours.	Ö	Ö	Ö	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 $\leq 6^{\circ}\text{C}$. 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.

8. SAMPLE COLLECTION PRESERVATION AND STORAGE

8.1. Samples are not chemically preserved.

8.2. Samples are stored at $4^{\circ}\text{C} \pm 2^{\circ}\text{C}$ in glass containers with Teflon®-lined caps.

8.3. Holding Times

8.3.1 Extraction is initiated within seven days of the sampling date for aqueous samples, 14 days for solid and waste samples.

8.3.2 For TCLP leachates, extraction is initiated within seven days from when the TCLP Leach tumbling has been completed, excluding the filtration 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 a LCS pair in place of a 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. Additional MS/MSD sets are included in the sample count.

9.4. Method Blank

- 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. 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. 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'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. On-going 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 - 9.4.4) but spiked with the LCS standard and the surrogates.

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.

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.

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.

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. If a new box of glass vials are used, then the steps are repeated to further ensure that variations due to glass vial size and shape are minimized. A log is kept of the lot number of the glass vials and the day the glass vials were prepared.

11. PROCEDURE

Procedures for separatory funnel liquid/liquid extraction (Section 11.2), continuous liquid/liquid extraction (Section 11.3), sonication extraction (Section 11.4), soxhlet extraction (Section 11.5), accelerated soxhlet (Section 11.6), waste dilution (Section 11.7), and extract concentration (Section 11.8).

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 shall 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.

Any deviations from this procedure identified after the work has been completed must be documented as a nonconformance, with a cause and corrective action described. A Nonconformance memo shall be used for this documentation.

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. If sample is a leachate (e.g. TCLP), compare the current pH against leachate log, note on the benchsheet if there is any discrepancy.
- 11.2.3. Measure the initial volume using 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. The LCS and/or LCSD and MS/MSD are spiked with the surrogate and matrix spike solutions, the method blank only with the surrogates.
- 11.2.5. Use 250 mL of leachate for TCLP pesticides and TCLP semivolatiles, measured in a beaker.
- 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 desired.
- 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. 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 H₂SO₄ or 10 N NaOH 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 2 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 should 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 should 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 H₂SO₄. 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 11.8 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 3 to 5 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. If sample is a leachate (e.g. TCLP), compare the current pH against leachate log. Note on the benchsheet if there is any discrepancy.
- 11.3.4. Measure the initial volume using 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. Use 1 L of reagent water for method blanks and LCS.

Use 250mL of leachate for TCLP 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 used in the leaching procedure 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 used in the leaching procedure 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.

- 11.3.5. Add reagent water to the extractor body until approximately 250 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 H₂SO₄ or 10 N NaOH 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°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 H₂SO₄. 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. Reattach the condenser and turn on 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 11.8 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 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 ± 0.2 g 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.
- 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 ± 0.2 g each, of parent sample for the MS and MSD samples.
- 11.4.7. Immediately add a minimum of 100 mL of solvent to the beaker.

Solvents:

Semivolatile GC/MS, TPH, Organochlorine pesticides and PCBs	1:1 v/v Methylene Chloride / Acetone

Note: Steps 11.4.5 - 11.4.9 should 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 *Microtip* 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 additional 100 mL minimum 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 with an additional 10 mL-20 mL of the methylene chloride/acetone 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 11.8 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. Soxhlet

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. 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 or Administrator).

- 11.5.3. Place approximately 200mL of solvent into a 250 mL flat bottom flask containing one or two clean boiling chips. Weigh 30 g of sample ± 0.2 g into a thimble or in a beaker, recording the weight to the nearest 0.01 g on the benchsheet. Sample weights less than 30 g but over 5 g may be used if the appropriate reporting limits can be met.
- 11.5.4. 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 should be approximately the weight of soil used in each sample.
- 11.5.5. Add the appropriate amount of surrogate and matrix spiking solution as indicated in Tables 3, 4, 5, and 6.
- 11.5.6. Add anhydrous sodium sulfate to each sample and mix well. The mixture should 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.
- 11.5.7. 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	1:1 v/v Methylene
Organochlorine pesticides and PCBs	Chloride / Acetone

- 11.5.8. Allow the extract to cool after the extraction is complete, then disassemble by gently twisting the soxhlet from the flask.
- 11.5.9. The sample is now ready for the concentration step (Section 11.8).
- 11.5.10. Cover with aluminum foil if the extract is not concentrated immediately. Refer to Section 11.8 for concentration.

11.6. Accelerated Soxhlet (Soxtherm Trade Name)

- 11.6.1. Remove surrogate and matrix spiking solutions from refrigerator and allow to return 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 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).
- 11.6.3. Weigh 30 g of sample ± 0.2 g into a beaker or wide-mouth jar, recording the weight to the nearest 0.01 g on the benchsheet. Add 30 g of anhydrous sodium sulfate to each sample, and mix well. The mixture should 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.
- 11.6.4. Sample weights less than 30 g, but over 5 g may be used if the appropriate reporting limits can be met.
- 11.6.5. 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 should be approximately the weight of soil used in each sample.
- 11.6.6. Add the appropriate amount of surrogate and matrix spiking solution as indicated in Tables 3, 4, 5, and 6.
- 11.6.7. 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:

Semivolatiles MS, PAH, TPH	1:1 v/v Methylene Chloride /
Semivolatiles GC PCB, PEST, OPP	Acetone
	1:1 v/v Hexane/Acetone

- 11.6.8. Upon completion of the program, remove the extract beaker from the unit, let cool, and dispose of the extracted sample.
- 11.6.9. 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.

- 11.6.10. Place culture tubes on nitrogen evap and reduce to approximately 1-2mL and add appropriate solvent according to the extraction performed to bring the sample to the correct final volume.

11.7. Waste Dilution

- 11.7.1. Remove surrogate and matrix spiking solutions from refrigerator and allow to return to room temperature.
- 11.7.2. 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 ± 0.01 g.
- 11.7.3. 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).
- 11.7.4. 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. 8015B waste dilutions are diluted to approximately 10mL with DCM and are placed on the nitrogen evaporation unit to reduce to a 2 mL final volume.
- 11.7.5. Cap and shake or vortex each extract.
- 11.7.6. The sample is now ready for analysis.

11.8 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.

11.8.1. Kuderna-Danish (KD) Method:

- 11.8.1.1. 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.
- 11.8.1.2. 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.

11.8.1.3 Place the KD apparatus on a water bath (90-98°C) so that the tip of the concentrator tube is submerged. The water level should 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.

11.8.1.4 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.

11.8.1.5. 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.

11.9. Nitrogen Evaporation to Final Concentration

11.9.1. Transfer the CT to the evaporation apparatus

11.9.2. 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 should 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.

12. DATA ANALYSIS AND CALCULATIONS

Not applicable

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 procedure for the determination of the method detection limit is given in TestAmerica North Canton QA Policy S-Q-003 and NC-QA-0021.

13.2. Initial Demonstration

- 13.2.1. 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 should contain all the analytes of interest. The spiking level should 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.)
- 13.2.2. Four aliquots of the QC check sample are analyzed using the same procedures used to analyze samples, including sample preparation.
- 13.2.3. Calculations and acceptance criteria for the QC check samples are given in the determinative SOPs.

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.

14. POLLUTION PREVENTION

- 14.1. Within the constraints of following the methodology in this SOP, use of organic solvents should be minimized.

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 Safety Manual 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 waste water 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 PCB's 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

Company Confidential & Proprietary

Edition, Final Update III (December 1996). Sections 3500B, 3510C, 3520C, 3540C, 3550B, 3600C, 3610B, 3620B, 3640A, 3650B, 3660B, AND 3665A.

16.1.2. TestAmerica North Canton Quality Assurance Manual (QAM), current version.

16.2. Associated SOPs and Policies, latest version

16.2.1. QA Policy, QA-003

16.2.2. Glassware Washing, NC-QA-0014

16.2.3. Statistical Evaluation of Data and Development of Control Charts, NC-QA-0018, current version.

16.2.4. Method Detection Limits and Instrument Detection Limits, NC-QA-0021, current version.

16.2.5. Supplemental Practices for DoD Project Work SOP, NC-QA-0016

16.2.6. Method Detection Limit Studies, CA-Q-S-006, current version.

16.2.7. CORP-GC-0001NC, Gas Chromatographic Analysis based on Method 8000B, 8021B, 8081A, 8082, 8151A, 8015B, and 615, current version.

16.2.8. CORP-MS-0001NC, GC/MS Analysis based on Method 8270C, current version.

16.2.9. NC-GC-0007, Analysis of Pesticides and PCBs by EPA Method 608, current version.

16.2.10. NC-MS-0003, GC/MS Semivolatile Organic Compounds Capillary Column Technique Based on EPA Method 625, current version.

16.2.11. Standards and Reagents, SOP NC-QA-0017.

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.

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 4 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

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
BNA	1,2,4-Trichlorobenzene	100

Type	Compounds	Conc. (µg/mL)
BNA	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

Type	Compounds	Conc. (µg/mL)
BNA	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
	Nitrobenzene	100
	N-Nitrosodi-n-propylamine	100
	Phenanthrene	100
	Pyrene	100
	1,2,4-Trichlorobenzene	100
	4-Chloro-3-methylphenol	100
	2-Chlorophenol	100

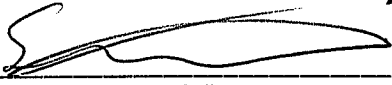
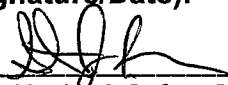
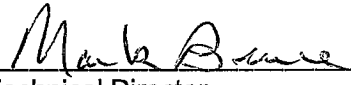
Type	Compounds	Conc. (µg/mL)
	2,4-Dichlorophenol	100
	2,4-Dimethylphenol	100
	2,4-Dinitrophenol	100
	2-Methyl-4,6-dinitrophenol	100
	2-Nitrophenol	100
	4-Nitrophenol	100
	Pentachlorophenol	100
	Phenol	100
	2,4,6-Trichlorophenol	100
	Acetophenone	100
	Atrazine	100
	Caprolactum	100
	Benzaldehyde	100
	1,1'-Biphenyl	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
	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

**Title: TOXICITY CHARACTERISTIC LEACHING PROCEDURE
AND SYNTHETIC PRECIPITATION
LEACHING PROCEDURE**

[SW846 Method 1311]

Approvals (Signature/Date):			
	3-18-08		3/18/08
Technology Specialist	Date	Health & Safety Coordinator	Date
	3/19/08		3/20/08
Quality Assurance Manager	Date	Laboratory Director	Date
	3/19/08		
Technical Director	Date		

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TABLE OF CONTENTS

1. SCOPE AND APPLICATION	3
2. SUMMARY OF METHOD.....	4
3. DEFINITIONS.....	5
4. INTERFERENCES.....	5
5. SAFETY.....	6
6. EQUIPMENT AND SUPPLIES.....	9
7. REAGENTS AND STANDARDS	10
8. SAMPLE COLLECTION, PRESERVATION AND STORAGE	11
9. QUALITY CONTROL	13
10. CALIBRATION AND STANDARDIZATION	14
11. PROCEDURE	14
12. DATA ANALYSIS AND CALCULATIONS	27
13. METHOD PERFORMANCE.....	29
14. POLLUTION PREVENTION	29
15. WASTE MANAGEMENT.....	29
16. REFERENCES.....	30
17. MISCELLANEOUS (METHOD INTERPRETATIONS, RECORD MANAGEMENT, ETC.).....	32

LIST OF APPENDICES

APPENDIX A - TABLE.....	33
APPENDIX B - FIGURES.....	35
APPENDIX C - LOGBOOK SHEETS.....	39
APPENDIX D - FLOW CHARTS.....	44

1. SCOPE AND APPLICATION

1.1. This SOP describes the application of the Toxicity Characteristic Leaching Procedure (TCLP), SW846 Method 1311. The Toxicity Characteristic (TC) of a waste material is established by determining the levels of 8 metals and 31 organic chemicals in the aqueous leachate of a waste. The TC is one of four criteria in 40 CFR Part 261 to determine whether a solid waste is classified as a hazardous waste. The other three are corrosivity, reactivity, and ignitability. The TC Rule utilizes the TCLP method to generate the leachate under controlled conditions that were designed to simulate leaching through a landfill. EPA's "worst case" waste disposal model assumes mismanaged wastes will be exposed to leaching by the acidic fluids generated in municipal landfills. The EPA's model also assumes the acid/base characteristics of the waste will be dominated by the landfill fluids. The TCLP procedure directs the testing laboratory to use a more acidic leaching fluid if the sample is an alkaline waste, again in keeping with the model's assumption that the acid fluids will dominate leaching chemistry over time.

1.2. The specific list of TC analytes and regulatory limits may be found in Appendix A.

Note: The list in Appendix A does not include the December 1994 EPA rule for Universal Treatment Standards for Land Disposal Restrictions. Those requirements include 216 specific metallic and organic compounds and, in some cases, lower detection limit requirements (see 40 CFR 268.40). TCLP leachates are part of the new Universal Treatment Standards, but the conventional analytical methods will not necessarily meet the new regulatory limits. Consult with the client and with TestAmerica Technical Specialists before establishing the instrumental methods for these regulations.

1.3. This SOP also describes the application of the Synthetic Precipitation Leaching Procedure (SPLP) which was designed to simulate the leaching that would occur if a waste was disposed in a landfill and exposed only to percolating rain water. The procedure is based on SW846 Method 1312. The list of analytes for SPLP may extend beyond the toxicity characteristic compounds shown in Appendix A. With the exception of the use of a modified extraction fluid, the SPLP and TCLP protocols are essentially equivalent. Where slight differences may exist between the SPLP and TCLP they are distinguished within this SOP.

1.4. The procedure is applicable to liquid, solid, and multiphase wastes.

1.5. The results obtained are highly dependent on the pH of the extracting solution, the length of time that the sample is exposed to the extracting solution, the temperature during extraction, and the particle size/surface area of the sample. These parameters must be carefully controlled.

- 1.6. The reporting limits are based on the individual samples as well as the individual analysis techniques. However, the sample is determined to be hazardous if it contains any analyte at levels greater than or equal to the regulatory limits.
- 1.7. If a total analysis of the waste demonstrates that individual analytes are not present in the waste, or they are present but at such low concentrations that the appropriate regulatory levels could not possibly be exceeded, the procedure need not be run. If the total analysis results indicate that TCLP is not required, the decision to cease TCLP analysis should be remanded to the client.
- 1.8. If an analysis of any one of the liquid fractions of the procedure leachate indicates that a regulated compound is present at such a high concentration that, even after accounting for dilution from the other fractions of the leachate, the concentration would be equal to or above the regulatory level for that compound, then the waste is hazardous and it may not be necessary to analyze the remaining fractions of the leachate. However, the remaining analyses should not be terminated without the approval of the client.
- 1.9. Volatile organic analysis of the leachate obtained using a bottle extraction, normally used for extractable organics and metals, can be used to demonstrate that a waste is hazardous, but only the ZHE option can be used to demonstrate that the concentration of volatile organic compounds is below regulatory limits due to potential analyte loss into the headspace during the bottle extraction.

2. SUMMARY OF METHOD

- 2.1. For liquid wastes that contain less than 0.5% dry solid material, the waste, after filtration through 0.6 to 0.8 μm glass fiber filter, is defined as the TCLP leachate.
- 2.2. For wastes containing greater than or equal to 0.5% solids, the liquid, if any, is separated from the solids and stored for later analysis or recombination with the leachate. The particle size of the remaining solid phase is reduced, if necessary. The solid phase is extracted with an amount of extraction fluid equal to 20 times the weight of the solid phase. For TCLP, the extraction fluid employed for extraction of non-volatile analytes is a function of the alkalinity of the solid phase of the waste. For SPLP, the extraction fluid employed is a function of the region of the country where the sample site is located if the sample is a soil. If the sample is a waste or wastewater the extraction fluid employed is a pH 4.2 solution. Two leachates may be generated: a) one for analysis of non-volatile constituents (semi-volatile organics, pesticides, herbicides and metals, and/or, b) one from a Zero Headspace Extractor (ZHE) for analysis of volatile organic constituents. Following extraction, the liquid leachate is separated from the solid phase by filtration through a 0.6 to 0.8 μm fiber filter.

- 2.3. If compatible (i.e., multiple phases will not form on combination), the initial liquid filtrate of the waste is added to the liquid leachate and these are prepared and analyzed together. If incompatible, the liquids are analyzed separately and the results are mathematically combined to yield a volume-weighted average concentration.

3. DEFINITIONS

- 3.1. “Leachate” is used to refer to the solutions generated from these procedures (TCLP, SPLP, deionized water leach).
- 3.2. “Wet Solids” is that fraction of a waste sample from which no liquid may be forced out by pressure filtration.

4. INTERFERENCES

- 4.1. Oily wastes may present unusual filtration and drying problems. If requested by the client and as recommended by EPA (see Figure 3), oily wastes can be assumed to be 100% liquid and analysis for total concentrations of contaminants will be performed. This applies specifically to samples containing viscous non-aqueous liquids that would be difficult to filter. Alternately, the oil may be subjected to pressure filtration. The portion that passes through the filter will be prepared and analyzed separately as an organic waste. The “wet solid” portion that remains behind on the filter will be subjected to leaching--prepared and analyzed. The results will then be mathematically combined.
- 4.2. Wastes containing free organic liquids (e.g., oil, paint thinner, fuel) usually require dilution prior to analysis to address the matrix interferences. In most instances this results in reporting limits elevated above the TCLP regulatory limits.
- 4.3. Solvents, reagents, glassware and other sample processing hardware may yield artifacts and/or interferences to sample analysis. All these materials must be demonstrated to be free from interferences under the conditions of the analysis by analyzing method blanks as described in Section 9 and the individual determinative SOPs.
- 4.4. Glassware and equipment contamination may result in analyte degradation. Soap residue on glassware and equipment may contribute to this. All glassware and equipment should be rinsed very carefully to avoid this problem.
- 4.5. Phthalates may be eliminated by proper glassware cleanup and by avoiding plastics. Only glass, Teflon or Type 316 stainless steel tumblers may be used for leachates to be analyzed for organics. Plastic tumblers may be used for leachates to be analyzed for the metals.
- 4.6. Overexposure of the sample to the environment will result in the loss of volatile components.

- 4.7. Potential interferences that may be encountered during analysis are discussed in the individual analytical methods.

5. SAFETY

- 5.1. Employees must abide by the policies and procedures in the Corporate Safety Manual and this document.
- 5.2. Gas pressurized equipment is employed in this procedure. Be sure all valves and gauges are operating properly and that none of the equipment, especially tubing, is over-pressurized. CAUTION: Do not open equipment that has been pressurized until it has returned to ambient pressure.
- 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 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
Acetic Acid	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.
Glacial Acetic Acid	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.
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.

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.
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.
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	2 mg/m ³ - Ceiling	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.
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 will be removed and discarded; other gloves will be cleaned immediately.
- 5.5. A rotary agitation apparatus is used in this procedure. Certain samples may break the glass jars used in the procedure. For these samples, extra caution, including plastic or polyethylene overwraps of the glass jar, may be necessary. Turning the jar or bottle sideways rather than tumbling end over end may also reduce the chance of breakage. If sideways tumbling is used, note this change in the logbook comment section. Guards must be placed in front of any rotating equipment.
- 5.6. Secure tumbler and extraction apparatus before starting rotary agitation apparatus.
- 5.7. During sample rotation, pressure may build up inside the bottle. Periodic venting of the bottle will relieve pressure.
- 5.8. Any cyanide containing waste or soil may result in the formation of hydrogen cyanide gas when exposed to acidic conditions. SPLP Fluid #3 reagent water must be used for these samples. **NOTE: Do not use an acidic SPLP fluid due to the potential release of hydrogen cyanide gas.**
- 5.9. Exposure to hazardous chemicals must be maintained as low as reasonably achievable; therefore, unless they are known to be non-hazardous, all samples must be opened, transferred and prepared in a fume hood, or under other means of mechanical ventilation, where possible. 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 will be kept closed unless transfers are being made.
- 5.10. The preparation of standards and reagents and glassware cleaning procedures that involve solvents such as methylene chloride will be conducted in a fume hood with the sash closed as far as the operation will permit.
- 5.11. 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.
- 5.12. Due to the potential for ignition, flammability or production of noxious fumes, do not attempt to dry non-aqueous liquid samples in an oven. Use extended drying in a ventilation hood.

6. EQUIPMENT AND SUPPLIES

- 6.1. Extraction vessels
 - 6.1.1. For volatile analytes - zero-headspace extraction (ZHE) vessel, gas-pressure actuated, Millipore YT3009OHW or equivalent (see Figure 2)
 - 6.1.2. For metals - either borosilicate glass jars (2.5 L, with Teflon lid inserts) or 2.5 L HDPE (Nalgene or equivalent) bottles may be used
 - 6.1.3. For non-volatile organics - only borosilicate glass may be used
- 6.2. Vacuum filtration apparatus and stainless steel pressure filtration apparatus (142 mm diameter), capable of 0 - 50 psi
- 6.3. Borosilicate glass fiber filters, 0.6 - 0.8 μm (Whatman GF/F 14.2 cm, 9.0 cm, 4.7 cm, 0.7 μm or equivalent). When analyzing for metals, wash the filters with 1 N nitric acid and de-ionized water prior to use, or purchase pre-washed filters. Glass fiber filters are fragile and should be handled with care.
- 6.4. Rotary agitation apparatus, multiple-vessel, Associated Design and Manufacturing Company 3740-6 or equivalent (see Figure 1). The apparatus must be capable of rotating the extraction vessel in an end-over-end fashion at 30 ± 2 rpm.
- 6.5. ZHE Extract Collection Device: Gas-tight syringes, 50 or 100 mL capacity, Hamilton 0158330 or equivalent
- 6.6. Top loading balance, capable of 0 - 4000 $\pm$ 0.01g (all measurements are to be within $\pm$ 0.1 grams)
- 6.7. pH meter and probe capable of reading to the nearest 0.01 unit, and with automatic temperature compensation
- 6.8. Magnetic stirrer/hotplate and stirring bars
- 6.9. VOA vials, 40 mL, with caps and septa
- 6.10. Glass bottles, 1 liter, with Teflon lid-inserts
- 6.11. Nalgene plastic bottles or equivalent, 1 liter
- 6.12. Miscellaneous laboratory glassware and equipment

7. REAGENTS AND STANDARDS

- 7.1. Reagent water for non-volatile constituents must be produced by a Millipore DI system or equivalent. For volatile constituents, water must be passed through an activated carbon filter bed (Milli-Q or tap water passed through activated carbon). Reagent water must be free of the analytes of interest as demonstrated through the analysis of method blanks.
- 7.2. Hydrochloric acid, 1 N: Carefully add 83 mL concentrated reagent grade HCl to 800 mL reagent water, cool and dilute to 1 liter with reagent water. Cap and shake to mix well.
- 7.3. Sodium hydroxide, 1 N: Carefully add 40 g reagent grade NaOH pellets to 800 mL reagent water, stir until the pellets are completely dissolved, cool and dilute to 1 liter with reagent water.

CAUTION: Heat is generated during this process.

- 7.4. Acetic acid, glacial: concentrated, reagent grade liquid (HOAc)
- 7.6. pH calibration solutions: buffered to a pH of 4, 7, and 10. Commercially available.
- 7.5. TCLP Leaching Fluids

7.5.1. General Comments

- 7.5.1.1. The pH of both solutions listed below should be monitored daily and the pH probes are to be calibrated prior to use.
- 7.5.1.2. The leaching fluids MUST be prepared correctly. If the desired pH range is not achieved and maintained, the TCLP may yield erroneous results due to improper leaching. If the pH is not within the specifications, the fluid must be discarded and fresh extraction fluid prepared.
- 7.5.1.3. Additional volumes of extraction fluids listed below may be prepared by multiplying the amounts of acetic acid and NaOH by the number of liters of extraction fluid required.
- 7.5.1.4. At the end of the day, all remaining buffer solutions must be properly discarded.
- 7.5.2. TCLP Fluid #1: Carefully add 5.7 mL glacial acetic acid and 64.3 mL of 1 N NaOH to 500 mL reagent water in a 1 liter volumetric flask. Dilute to a final volume of 1 L with reagent water, cap and shake to mix well. For 8 L of fluid use

45.6 mL glacial acetic acid and 514 mL 1N NaOH, dilute to 8 L with reagent water. When correctly prepared, the pH of this solution is 4.93 ± 0.05 . The density of TCLP fluid #1 is 0.997 g/mL.

- 7.5.3. TCLP Fluid #2: Carefully add 5.7 mL glacial acetic acid to 500 mL reagent water in a 1 liter volumetric flask. Dilute to a final volume of 1 L with reagent water, cap and shake to mix well. For 8 L of fluid use 45.6 mL glacial acetic acid, dilute to 8 L with reagent water. When correctly prepared, the pH of this solution is 2.88 ± 0.05 . The density of TCLP fluid #2 is 0.997 g/mL.
- 7.6. Nitric acid, 50% solution: Slowly and carefully add 500 mL concentrated HNO_3 to 500 mL reagent water. Cap and shake to mix well.
- 7.7. Sulfuric acid / nitric acid (60/40 weight percent mixture) $\text{H}_2\text{SO}_4/\text{HNO}_3$. Cautiously mix 60 g of concentrated sulfuric acid with 40 g of concentrated nitric acid.
- 7.8. SPLP Leaching fluids
- 7.8.1. SPLP solutions are unbuffered and exact pH may not be attained. The pH of TCLP and SPLP fluids should be checked prior to use. If not within specifications, the fluid should be discarded and fresh fluid prepared.
- 7.8.2. SPLP fluid #1: Add 60/40 weight percent mixture of sulfuric and nitric acids to reagent water until the pH is 4.20 ± 0.05 . This fluid is used for soils from a site that is east of the Mississippi River and for wastes and waste waters.
- 7.8.3. SPLP fluid #2: Add 60/40 weight percent mixture of sulfuric and nitric acids to reagent water until the pH is 5.00 ± 0.05 . This fluid is used for soils from a site that is west of the Mississippi River.
- 7.8.4. SPLP fluid #3: This fluid is reagent water and is used for leaching of volatiles. Additionally, any cyanide-containing waste or soil is leached with fluid #3 because leaching of cyanide containing samples under acidic conditions may result in the formation of hydrogen cyanide gas.
- 7.9. Methanol and methylene chloride - used to aid in cleaning oil contaminated equipment.

8. SAMPLE COLLECTION, PRESERVATION AND STORAGE

- 8.1. Samples being analyzed for non-volatile organic compounds should be collected and stored in glass containers with Teflon lid liners. Chemical preservatives shall NOT be added UNTIL AFTER leachate generation. Undamaged glass containers should be washed according to SOP NC-QA-0014.

- 8.2. Samples being analyzed for metals only can be collected in either glass or polyethylene containers.
- 8.3. When the waste is to be evaluated for volatile analytes, care should be taken to minimize the loss of volatiles. Samples shall be collected and stored in a manner intended to prevent the loss of volatile analytes (e.g., samples should be collected in Teflon lined septum capped vials with minimal headspace and stored at $4 \pm 2^{\circ}\text{C}$). Samples should be opened only immediately prior to extraction.
- 8.4. Samples should be refrigerated to $4 \pm 2^{\circ}\text{C}$ unless refrigeration results in irreversible physical changes to the waste. If precipitation occurs, the entire sample (including precipitate) should be extracted.
- 8.5. The minimum TCLP sample collection size is determined by the physical state or states of the waste and the analytes of concern. The amount of waste required varies with the percent solids. The lower the percent solids, the more waste will be required for preliminary and final testing. For aqueous samples containing between 0.5 and 10% solids, several kilograms of sample are required to complete the analyses. The general minimal requirements when the samples are 100% solids include: 1 - 32 oz jar for semi-volatile organic analysis and metals, and 1 - 4 oz jar for volatile organic analysis. Low density sample materials, such as rags or vegetation, will require larger volumes of sample. For liquid samples (less than 50% solids), minimum requirements are 2 - 32 oz jars for semi-volatile organic analysis and metals, and 2 - 8 oz jars for volatile organic analysis. If volatile organic analysis is the only requested parameter, 2 separate jars are required. If matrix spike or duplicate control samples are requested, additional sample volume is required. If sufficient sample volumes were not received, analyses cannot be started and the client should be notified as soon as possible.
- 8.6. TCLP leachates should be prepared for analysis and analyzed as soon as possible following extraction. Leachates or portions of leachates for metallic analyte determinations must be acidified with nitric acid to a pH less than 2, unless precipitation occurs. If precipitation occurs upon addition of nitric acid to a small aliquot of the leachate, then the remaining portion of the leachate shall not be acidified and the leachate shall be analyzed as soon as possible. All other leachates should be stored under refrigeration ($4 \pm 2^{\circ}\text{C}$) until analyzed. ZHE leachates must be stored in VOA vials filled to eliminate all headspace.
- 8.7. Samples are subject to appropriate treatment within the following time periods.

Table 1
Holding Times (days)

Parameter	Collection to Start of TCLP Leach	End of TCLP Tumble to Preparation	Start of TCLP Leach or Semi- volatile Prep Extraction to Analysis	Total Elapsed Time
Volatiles	14	N/A	14	28
Semi-volatiles	14	7	40	61
Mercury	28	N/A	28	56
Other Metals	180	N/A	180	360

NOTE: The initial holding time is measured from date of collection to date TCLP extraction started. (This should be the TCLP extraction date in QuantIms.) Semi-volatile method prep holding time is measured from the day tumbling is complete to the start of method extraction. Subsequent analysis holding times are measured from the date extraction (TCLP or method prep) starts. If sample holding times are exceeded, the values obtained will be considered minimal concentrations. Exceeding holding times is not acceptable in establishing that a waste does not exceed the regulatory level. Exceeding the holding time will not invalidate characterization if the waste exceeds the regulatory limit. The Total Elapsed Time is to be used as guidance. If preps are initiated at the last possible moment of a holding time, the elapsed times may be exceeded.

9. QUALITY CONTROL

- 9.1. Quality Control Batch (QC Batch) - QA-003 defines a QC Batch as a set of up to 20 field samples of similar matrix that behave similarly and are processed using the same procedures, reagents and standards within the same time period. The same lot of reagents must be used within a batch. A minimum of one TCLP extraction blank (Method Blank), one Laboratory Control Sample (LCS), one Matrix Spike (MS), and one Matrix Spike Duplicate (MSD) will be prepared with each TCLP leachate batch.
- 9.2. TCLP Extraction Blanks - A minimum of one blank (using the same extraction fluid as used for the samples) must be prepared and analyzed for every batch of samples extracted in a particular vessel type. The blanks are generated in the same way as the samples (i.e., blanks will be tumbled and filtered with the samples). If particle size reduction was performed on any sample in the batch, an equipment blank will be generated by passing blank fluid through the particle reduction apparatus. ZHE Extraction vessels will be uniquely numbered. Consult the TestAmerica QC Program and the individual analysis SOPs for blank acceptance criteria.
- 9.3. Laboratory Control Sample (LCS) - A LCS is required with each batch of 20 or fewer samples. The LCS shall be generated after a batch of TCLP leachates have been

generated (i.e., at the time of the preparative digestion or extraction) by spiking an aliquot of the appropriate extraction fluid used for that batch or reagent water. Consult the individual analysis SOPs for additional LCS guidance (i.e., spike amounts, spike levels, recovery criteria, etc.).

9.4. Matrix Spike (MS/MSD) - Matrix spikes are used to monitor the performance of the analytical methods on the matrix and to assess the presence of interferences. A MS/MSD pair are required with each batch of 20 or fewer samples.

9.4.1. Matrix spikes are to be added after filtration of the TCLP leachate. Spikes are not to be added prior to the TCLP leaching. For metals, matrix spikes are to be added before preservation with nitric acid.

9.4.2. Consult the individual analysis SOPs for additional guidance on spike compounds and levels.

9.5. Corrective Actions

9.5.1. Consult the TestAmerica QC Program and individual analysis SOPs for corrective action for blanks and LCS

9.5.2. Method of Standard Additions (MSA) shall be used for mercury if all of the following conditions are met:

- Recovery of the analyte in matrix spike is not at least 50%,
- The concentration of the analyte does not exceed the regulatory level, and
- The concentration of the analyte measured in the sample is within 20% of the appropriate regulatory level.

If the matrix spike recovery is 5% or less due to dilution or matrix interference, contact the project manager and client for guidance. The client should also be contacted prior to initiation of any MSA steps. Refer to the individual analysis SOPs for details on how to perform MSA analysis.

10. CALIBRATION AND STANDARDIZATION

10.1. Refer to appropriate analysis SOPs.

11. PROCEDURE

11.1. General Comments

11.1.1.1. 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 in procedure shall be completely documented on a Nonconformance Memo kept in the project file and described in the final report. The variation must be approved by a Project Manager, Technical Specialist, and QA Manager. Any unauthorized deviations from this procedure must also be documented as a nonconformance with a cause and corrective action described.

11.1.1.2. All masses should be recorded to the nearest 0.1 g.

11.2. Preliminary Sample Evaluations (Refer to Flow Chart #1, Appendix D)

11.2.1. Determine the total volume of TCLP leachate (solid phase leachate plus liquid filtrate) that needs to be generated for analysis according to the following:

Table 2
Recommended TCLP Leachate Volume

Analysis	TCLP Required Volume (mL)	SPLP Required Volume (mL)
Volatiles	3 x 40	3 x 40
Semi-volatiles	500	1000
Pesticides	500	1000
Herbicides	500	1000
Metals	300	300

11.2.1.1. For TCLP and SPLP samples used for matrix spike and matrix spike duplicate analysis, two to three times the listed volumes are required.

11.2.2. Sample Description (determine sample matrix)

11.2.2.1. Solid - If the waste will obviously yield no free liquid when subjected to pressure filtration, then proceed to Section 11.2.5 or 11.4 (Bottle Extraction Procedure or ZHE Procedure).

11.2.2.2. Liquid - If the sample is a monophasic liquid, proceed to Section 11.2.3 (Percent Solid Determination).

11.2.2.3. Multiphasic – The sample has discernible layers (liquid/liquid or liquid/solid). If more than one container of multi-phasic materials is received from the field, each container might show different amounts of each phase. Consult client to determine sample selection alternatives (composite all sample containers, select one, resample, etc.) if this occurs.

11.2.3. Solids Determination

11.2.3.1. Determine Type of Filtration Apparatus and Process

11.2.3.1.1. Percent Solids and ZHE Extractions - The ZHE filtration apparatus cannot accurately determine percent solids less than 5%. If an extraction is to be performed solely for volatile organic compounds and the percent solids concentration is apparently greater than 5%, proceed to Section 11.4 (ZHE Extraction Procedure, Volatile Constituents). Otherwise, continue with Section 11.2.3.2. The aliquot of sample used here cannot be used again for the ZHE extraction.

11.2.3.1.2. If the sample is mostly a non-viscous liquid (water or non-viscous organic liquid) of low solids content (expected to be < 0.5%) , vacuum filtration should be used initially. Proceed to determination of percent dry solids (Section 11.2.3.2)

11.2.3.1.3. If the sample is viscous (sludge, oil, or is expected to have solids content > 0.5%), use pressure filtration. Proceed to determination of wet solids (Section 11.2.3.3).

11.2.3.2. Determination of percent dry solids

11.2.3.2.1. Measure and record the weight of the filter. Load the filter into the filter holder and assemble vacuum filter apparatus.

11.2.3.2.2. Homogenize the waste, then transfer 100 g subsample to a glass beaker. Record the sample weight in the percent dry solids section of the logbook.

11.2.3.2.3. Turn on vacuum source. Transfer the sample to the vacuum filtration device attempting to spread the waste sample evenly over the surface of the filter. Be sure to transfer all

particulates from the beaker to the filter. Use a reagent water rinse if necessary.

- 11.2.3.2.4. Once all liquid has been pulled through the filter, remove the filter with the wet solids from the vacuum filtration apparatus.
- 11.2.3.2.5. Dry the filter and solid phase at 100 ± 20 ° C for approximately 15 minutes.
- 11.2.3.2.6. Remove the filter from the oven, and allow to cool in a desiccator.
- 11.2.3.2.7. Weigh and record the dry weight of filter + particulates.
- 11.2.3.2.8. Calculate and record the percent dry solids.
- 11.2.3.2.9. If the percent dry solids is $\geq 0.5\%$, repeat the drying step. Weigh and record the second filter + particulates dry weight. If the two weighings do not agree within 1%, perform additional drying and weighing until successive weighings agree within 1%.
- 11.2.3.2.10. If the dry solids result is $\geq 0.5\%$, proceed to Section 11.2.3.3 using a fresh wet portion of the multiphase waste.
- 11.2.3.2.11. If the percent solids result is less than 0.5%, discard the solid phase. No leaching will be necessary. Filter sufficient sample with either the pressure filtration system or ZHE system as described in Sections 11.3 and 11.4. The filtrate is the TCLP leachate.

11.2.3.3. Determination of wet solids

- 11.2.3.3.1. Assemble the pressure filtration apparatus (use blunt forceps to handle the 0.6 to 0.8 μ m filter membrane).
- 11.2.3.3.2. Homogenize the waste, transfer a minimum of a 100 mL subsample to the glass beaker. Measure and record the gross weight (logbook Column A).
- 11.2.3.3.3. Measure and record the tare weight of the filtrate collection bottle (logbook Column D).

- 11.2.3.3.4. Transfer the sample to the filtration device attempting to spread the waste sample evenly over the surface of the filter. Measure and record the tare weight of the empty glass beaker and any residual sample (logbook Column B).
- 11.2.3.3.5. Calculate and record the net weight of sample used for testing (logbook Column C).
- 11.2.3.3.6. Slowly apply gentle pressure of 10 psi to the filtration apparatus. Allow the sample to filter until no SIGNIFICANT additional liquid has passed through the filter during a two-minute period.
- 11.2.3.3.7. If necessary, repeat previous step by increasing the pressure in 10 psi increments until a maximum of 50 psi is reached. Stop the filtration when no additional filtrate is generated within a two-minute period.

Note: Some samples will contain liquid material that does not filter (e.g., oil). Do not attempt to filter the sample again by exchanging filters. Viscous oils, or any wastes which do not pass through the filter, are classified by the method as a solid.
- 11.2.3.3.8. Remove the filtrate collection bottle, weigh and record the gross weight (logbook Column E).
- 11.2.3.3.9. Calculate and record the net weight of filtrate (logbook Column F). This result will be used in the percent solids calculation.
- 11.2.3.3.10. To determine the amount of filtrate, place the exact same type and size container as the filtrate container next to the filtrate. Add water to the exact level as the filtrate container to the empty container. Transfer the water to a graduated cylinder and record the volume. This step will reduce the amount of contamination, which may exist from transferring the filtrate to a graduated cylinder.
- 11.2.3.3.11. Retain the filtrate for possible recombination with the leachate in Section 11.3.7. Retain the filter and wet solids for the leaching in Section 11.3.

- 11.2.3.3.12. For multiphase sample preparations, calculate the total weight of wet solids and record the result in logbook Column G.

11.2.4. Particle-size Reduction for Fluid Selection

- 11.2.4.1. The subsample used for fluid selection must consist of particles less than approximately 1 mm in diameter (versus the less than 1 cm requirement for the material used for the actual extraction). The method requires a smaller particle size to partially compensate for the shorter duration of contact time with the leachate solution as compared to the full extraction. Inappropriate use of coarser materials could result in the selection of the wrong fluid type.
- 11.2.4.2. Surface area exclusion - size reduction is not required if the sample surface area is greater than or equal to 3.1 cm² per gram.
- 11.2.4.3. If the sample contains particles greater than approximately 1 mm in diameter, crush, cut, or grind the solids to the required size.
- 11.2.4.4. Consult a supervisor or manager when dealing with unusual sample matrices (e.g., wood, cloth, metal, brick).

11.2.5. Determination of Appropriate Extraction Fluid

- 11.2.5.1. If the solid content is greater than or equal to 0.5%, and if the sample is being analyzed for metals or nonvolatile organic compounds, the type of leaching solution must be determined.
- 11.2.5.2. Follow times, temperature, and particle size specified in this section as closely as possible. If reaction time between the acid solution and solid waste is too short or too long, the procedure may produce false pH readings.
- 11.2.5.3. For SPLP, refer to Section 7.8 for fluid selection. Record the fluid type in the logbook.
- 11.2.5.4. The TCLP leaching fluid for all volatiles is TCLP Fluid #1.
- 11.2.5.5. TCLP leach fluid determination for non-volatile analytes
- 11.2.5.5.1. Calibrate the pH meter with fresh buffer solution in accordance with the pH SOP.

11.2.5.5.2. Weigh out a 5.0 ± 0.1 g subsample (less than 1 mm particle size) of the solid phase into a glass or plastic container, and record in the logbook. Note: If sample quantity is limited, consult supervisor or manager.

Note: Many multiphase samples have limited solids quantity . In these instances, use a 5 g aliquot of the whole sample. Document this difference in the logbook comment section.

11.2.5.5.3. Add 96.5 ± 1.0 mL of reagent water, add magnetic stir bar, cover with a watchglass, and stir for 5 minutes.

11.2.5.5.4. Measure and record the pre-test sample pH in the logbook.

Note: To avoid damaging a glass pH probe when organic liquid is present, use narrow range pH indicator paper or an ISFET pH meter.

11.2.5.5.5. If the pH is less than or equal to 5.0, use TCLP Fluid #1.

11.2.5.5.6. If the fluid pH is greater than 5.0, add 3.5 mL 1 N HCl. Slurry the sample briefly. Insert therm into room temp DI water in one vial in each pre-test sample group to monitor the temperature. All samples in the group must be heated at the same time in order for the temperature of the one monitored sample to represent the others. Heat to $50 \pm 2^{\circ}\text{C}$ and maintain for ten minutes.

Note: The heating cycle is a critical step. If the solid waste does not remain in contact with the acidic solution under specified time and temperature conditions, an erroneous pH may be measured.

11.2.5.5.7. Cool to room temperature.

11.2.5.5.8. Measure and record the pH immediately after the sample has reached room temperature.

11.2.5.5.8.1. If the pH is less than or equal to 5.0, use TCLP Fluid #1. Record the buffer in the logbook.

11.2.5.5.8.2. If the pH is greater than 5.0, use TCLP Fluid #2. Record the buffer in the logbook.

11.2.6. For samples requiring analysis for semi-volatile organics, pesticides, herbicides or metals proceed to Section 11.3.

11.2.7. For samples requiring analysis for volatile organics (ZHE), proceed to Section 11.4.

11.3. Bottle Extraction Procedure: Non-Volatile Constituents: Semi-Volatiles, Pesticides, Herbicides, Metals (Refer to Flow Chart #2, Appendix D)

11.3.1. Evaluate the solid portion of the waste for particle size. If it contains particles greater than 1 cm in size, prepare the solid portion of the waste for leaching by crushing, cutting, or grinding such that all particles are less than 1 cm in size (i.e., capable of passing through a 9.5 mm, 0.375 inch, standard sieve). Size reduction is not required if the sample surface area is greater than or equal to 3.1 cm² per gram. If particle size reduction was required, record this in comments column in logbook.

11.3.1.1. Consult your supervisor or manager when dealing with unusual sample matrices (e.g., wood, cloth, metal, brick). Scissors or shears may be used to cut cloth, plastic or sheet metal. Saws may be used for wood or solid metal. Determination of particle size reduction tools should take into account the requested analytes (e.g. avoid chromium steel tools when TCLP metals have been requested). Bricks, rocks, or other solids amenable to grinding may be subcontracted out for particle size reduction (contact PA or PM). Note that size reduction to fine powder is not appropriate, and could invalidate results. If necessary, consult client for guidance.

11.3.2. Determine the minimum total volume of solid phase leachate that needs to be generated. Refer to Section 11.2.1.

11.3.3. Use 100 g of solid unless sample quantity is limited. If limited sample, divide the total volume of solid phase leachate required by 20 to determine the minimum mass of solid phase required for leaching. Round this mass UP to the nearest 5g. Client must be notified if less than 100 g of solid material is used.

Note: Solid phase material is often in limited quantity from multiphase samples. Generally all the *solid* phase material and the filter from Section 11.2.3.3.11 are transferred to the leaching bottle.

- 11.3.4. All non-ZHE extraction vessels should be uniquely numbered. If breakage occurs, replacement vessels must be numbered with the original vessel identification number prior to use. Breakage must be noted in the logbook comments identifying the affected sample and vessel number.
- 11.3.5. Weigh the required mass of solid phase into an appropriate extraction vessel (plastic for metals only, glass for all others) and **slowly** add 20 times its mass of appropriate leaching fluid (e.g., 100 g of sample would require 2000 mL of leaching fluid). Record the weight of the sample aliquoted for the extraction. Record the volume of extraction fluid added in the logbook if other than 2000 mL. Record the pH.
- 11.3.6. Ensure any effervescence has stopped before capping the bottle tightly. Secure in a rotary agitator and rotate end-over-end at 28-32 rpm for 16-20 hours. The temperature of the room should be $23 \pm 2^{\circ}\text{C}$. Record the rotary agitator I.D. and the date and time extraction is started and completed in the logbook.
- Note:** As agitation continues, pressure may build up within the bottle for some types of wastes. To relieve excessive pressure, the bottle may be removed and opened periodically in a properly vented hood to relieve any built-up pressure.
- 11.3.7. After tumbling in the rotary agitator is completed, remove the bottle and allow the solids to settle. Record the date and time the extraction is completed in the logbook. If sample was multiphase with an initial filtrate, drop a few drops of the filtrate (with a disposable glass pipette) into the extraction bottle and observe whether the filtrate is insoluble or forms a precipitate with the leachate. If so then the filtrate is not compatible with the leachate and must be bottled and analyzed separately. The results are normally mathematically recombined (Section 12.1.2). If the filtrate is compatible with the leachate (i.e., completely soluble) then pour the entire filtrate into the leachate bottle, recap and mix. Proceed with the leachate filtration step in the next section.
- 11.3.8. Filter the sample using pressure filtration by filtering through a new glass fiber filter. For final filtration of the TCLP leachate, the glass fiber filter may be changed, if necessary, to facilitate filtration. Filters must be acid washed if metals are to be determined (see Section 6.3). The entire sample need not be filtered; however, sufficient volume should be generated to support the required analyses.
- 11.3.9. Measure the pH of the TCLP leachate and record in the logbook. (Use narrow range pH paper or ISFET pH meter to measure the pH of oily samples as a glass pH probe may be damaged.)

11.3.10. Prepare sufficient volume for MS/MSD quality control testing. Refer to the appropriate determinative SOPs for further guidance on the spike components, levels and action criteria.

11.3.11. Immediately preserve the leachate as follows:

Metals pH < 2 w/ HNO₃ for aqueous filtrates and leachates
(do not acidify oils and other non-aqueous liquids)

All others Refrigerate to 4 ± 2 °C

Note: Refer to Section 8.6 if precipitation occurs upon preservation.

11.3.12. Label each sample with the appropriate information and submit to the appropriate analytical groups for prep and analysis. For multiphase samples requiring mathematical recombination provide copies of the TCLP preparation logbook sheets to the sample preparation and analysis groups. Most mathematically recombined samples will require data entry for the filtrate and leachate portions as well as for the mathematically recombined results. Contact the Project Manager to ensure the proper sample login is completed.

11.4. ZHE Extraction Procedure: Volatile Constituents (Refer to Flow Chart #3, Appendix D)

11.4.1. Use the ZHE device to obtain a TCLP leachate for analysis of volatile compounds only. Leachate resulting from the use of the ZHE shall NOT be used to evaluate the mobility of non-volatile analytes (e.g., metals, pesticides, herbicides and semi-volatile organics).

11.4.2. Due to some shortcomings of the method, losses of volatile compounds may occur. Extra care should be observed during the ZHE procedure to ensure that such losses are minimized. Charge the ZHE with sample only once and do not open the device until the final extract has been collected. Do not allow the waste, the initial liquid phase, or the extract to be exposed to the atmosphere any longer than necessary.

11.4.3. Install new O-rings and adjust the ZHE piston in the ZHE body to the appropriate height (slightly moisten the O-rings with leaching fluid if necessary).

11.4.4. If the preliminary evaluations indicated the need for particle size reduction, homogenize the waste, weigh out a sufficient size subsample and prepare for leaching by crushing, cutting, or grinding such that all particles are less than 1 cm in size as measured with a ruler (Do NOT sieve the sample). Size

reduction is not required if the sample surface area is greater than or equal to 3.1 cm² per gram. If particle size reduction was required, record this in the comments column of the logbook.

Note: To minimize loss of volatiles, samples for volatiles that require particle size reduction should be kept in sample storage (at 4°C) until immediately before size reduction. Aggressive reduction which would generate heat should be avoided and exposure of the waste to the atmosphere should be avoided to the extent possible. Size reduction to a fine powder is not appropriate. Also see Section 11.3.1.

11.4.4.1. Consult your supervisor or manager when dealing with unusual sample matrices (e.g., wood, cloth, metal, brick). Scissors or shears may be used to cut cloth, plastic or sheet metal. Saws may be used for wood or solid metal. Bricks, rocks, or other solids amenable to grinding may be subcontracted out for particle size reduction (contact PM).

11.4.5. Homogenize and transfer an appropriate size subsample of the waste into the ZHE and record the mass in the logbook.

11.4.5.1. For wastes that are solid, a 25 g sample is used.

11.4.5.2. For wastes containing < 0.5% solids, the liquid portion of the waste, after filtration, is defined as the TCLP leachate. Filter enough of the sample to support all of the volatile analyses required.

11.4.5.3. If the sample has ≥ 0.5% solids and has non-volatile TCLP/SPLP requested, the appropriate sample size should be estimated based on the wet solids content determined in Section 11.2.3.3. If ZHE only, use visual wet solids estimate to sample subaliquot.

Note: For wastes containing greater than 0.5% wet or dry solids, the “solids” value from the ZHE filtration process may be used to determine the volume of fluid to load into the ZHE. This approach is recommended since the solids value from Section 11.2.3.3 may differ from the ZHE filtration solids due to sample variability or differences in the filtration apparatus.

11.4.6. Carefully place the glass fiber filter between the support screens and secure to the ZHE. Tighten all the fittings.

11.4.7. Place the ZHE in a vertical position; open both the gas AND liquid inlet/outlet valves. Attach a gas line to the gas inlet/outlet valve.

- 11.4.8. If the waste is solid, slowly increase the pressure to a maximum of 50 psi to force out as much headspace as possible and proceed to Section 11.4.13.
- 11.4.9. If this is a mutliphase sample, carefully apply gentle pressure of 10 psi (or more, if necessary) to force all headspace slowly out of the ZHE. At the FIRST appearance of liquid from the liquid inlet/outlet valve, quickly close the valve and discontinue gas pressure.
- 11.4.10. Assemble a syringe and place the plunger in all the way. Attach the pre-weighed syringe to the liquid inlet/outlet valve and open the valve. Record the tare weight of the collection syringe in Column D of the logbook. .
- 11.4.11. Carefully apply gas pressure of no more than 10 psi to force out the liquid phase. Allow the sample to filter until no SIGNIFICANT additional filtrate has passed in a two-minute period.

Note: If the capacity of the syringe is reached, close the liquid inlet/outlet valve, discontinue gas pressure, remove the syringe, weigh, record weight in Column E and filtrate volume in the logbook. Return to Section 11.4.10.

- 11.4.12. Repeat previous step increasing the pressure in 10 PSI increments until 50 psi is reached and no significant liquid has passed in a 2 minute period. Close the valve and discontinue gas pressure. Remove the collection device and record the total weight of the collection device with filtrate in column E and filtrate volume in the logbook. Transfer the filtrate to VOA vials and label appropriately. Calculate the weight of filtrate collected and record in Column F in the logbook.

Note: If the original waste contained less than 0.5% solids (Section 11.2.3.2), this filtrate is defined as the TCLP leachate and you may proceed to Section 11.4.22. Otherwise, save the vials by storing at 4°C under minimal headspace conditions, for recombination as in Section 11.4.21.

The material remaining in the ZHE is defined to be the “solid” phase. Calculate the weight of the solid phase and record in Column G of the logbook by subtracting the weight of the filtrate from the weight of the sample.

- 11.4.13. Determine the amount of buffer to use. Solid samples use 500 mL of leach fluid (20 X 25 g). For multiphase samples use the wet solids (Column G) amount and multiply by 20. Record the leach fluid volume in Column H of the logbook.

Note: The TCLP ZHE prep uses only TCLP fluid #1; the SPLP ZHE prep uses only SPLP fluid #3.

- 11.4.14. Load the fluid transfer reservoir with an excess of Fluid #1 and preflush the transfer line to eliminate air pockets. Be sure the required volume remains.
- 11.4.15. Attach the transfer line to the liquid inlet/outlet valve and open the valve. Carefully pump the required volume into the ZHE and close the valve. Disconnect the transfer line.
- 11.4.16. Check the ZHE to make sure all the valves are closed and manually rotate the ZHE (end-over-end) two or three times. Reposition the ZHE in the vertical position.
- 11.4.17. Pressurize the ZHE to 5-10 psi. If the ZHE appears to be leaking, follow the corrective action protocols recommended by the manufacturer and repeat the analysis.
- 11.4.18. Slowly open the liquid inlet/outlet valve to bleed out any headspace that may have been introduced during the introduction of the Fluid. Upon the first sign of liquid from the valve, close the valve.
- 11.4.19. Repressurize the ZHE to 5-10 psi and place in the rotary agitator. Rotate at 28-32 rpm for 16-20 hours. Room temperature should be 23 ± 2 °C. The room temperature is recorded using a continuous temperature monitor.
- 11.4.20. Confirm that the pressure of 5-10 psi was maintained throughout the leaching. If it was NOT maintained, return to Section 11.4 and repeat the leachate with a new aliquot of sample.
- 11.4.21. If there is an initial liquid filtrate (Sec 11.4.12) determine if it is compatible with the leachate if the filtrate has not been previously tested (Sec. 11.3.7).
 - 11.4.21.1. Remove the plunger from the syringe and attach the barrel to the ZHE vessel. Open the outlet valve and pressurize as necessary to transfer about 1 mL of leachate into the syringe. Close the outlet valve.
 - 11.4.21.2. With a glass pipette transfer a few drops of initial filtrate into the open syringe barrel. Formation of separate layers or a precipitate indicates the filtrate and leachate are not compatible. Bottle the filtrate for separate preparation and analysis. The results are normally mathematically recombined.
 - 11.4.21.3. If the filtrate is compatible gently pour the remainder of the filtrate into the syringe barrel. Install the plunger. Bleed any pressure in the

ZHE piston. Open the inlet/outlet valve and depress the syringe plunger to inject the filtrate into the ZHE vessel. Do not inject the air bubble (if present) from the syringe.

11.4.21.4. Close the valve and rotate a few times to mix. Proceed with leachate filtration as described in the next section.

11.4.22. Attach an empty syringe to the outlet valve. Open the valve and pressurize the piston to expel the leachate from the ZHE vessel. Following collection, store the TCLP leachate in 2 or 3 40-mL VOA vials with minimal headspace at 4 ± 2 °C and prepare for analysis as soon as possible using the appropriate organic analysis procedure (see Section 16.3).

11.4.23. If the individual phases are analyzed separately, combine the results mathematically by using the recombination calculation in Section 12.1.2. Provide copies of the TCLP preparation logbook sheets to the sample preparation and analysis groups. Most mathematically recombined samples will require data entry for the filtrate and leachate portions as well as for the mathematically recombined results. Contact the project manager to ensure the proper sample login is completed.

11.4.24. ZHE Vessel Cleaning

11.4.24.1. Disassemble the vessel.

11.4.24.2. Clean all parts (vessel, lid, bottom, piston, and metal filters) with soapy water.

11.4.24.3. Rinse all parts with tap water followed by D.I. water.

11.4.24.4. Discard all used gaskets.

12. DATA ANALYSIS AND CALCULATIONS

12.1. Calculations

12.1.1. Calculation of weight of extraction fluid to use:

Volume of extraction fluid = 20 X weight of wet solids to be extracted

12.1.2. Mathematical recombination of analytical results:

$$\text{Final Analyte Concentration} = \frac{(V_1 \times C_1) + (V_2 \times C_2)}{V_1 + V_2}$$

Where,

V_1 = total volume of the initial filtrate phase (L).

C_1 = analyte concentration in initial filtrate phase (mg/L).

V_2 = volume of the theoretical solid phase leachate (L).

C_2 = analyte concentration in solid phase leachate (mg/L),

12.2. Reporting Requirements

12.2.1. Follow these reporting conventions for multi-phase samples.

- 12.2.1.1. If both phases have positive results, use the values from each phase to calculate the recombined result. Use the reporting limit for each phase to calculate the recombined reporting limit.
- 12.2.1.2. If both phases are “ND” (not detected) the recombined result is “ND,” and the reporting limit is calculated from the reporting limit for each phase.
- 12.2.1.3. If one phase is “ND” and the other phase has a positive result, use the zero for the “ND” phase and the positive value for the other phase to calculate the combined result. This will produce a minimum known concentration. Alternatively, at client request, the maximum possible concentration can be calculated by using the reporting limit for the “ND” phase rather than zero. The combined reporting limit is based on the reporting limit for both phases

12.2.2. Units - regardless of the nature of the sample, all TCLP and SPLP results are reported in units of mg/L.

12.2.3. For limits and significant figures, consult the appropriate analytical methods (Section 16.3).

12.2.4. Anomalies - all anomalies observed during the leach procedure must be noted on the worksheet or an NCM form. Some examples of such anomalies are:

- 12.2.4.1. Sample was monolithic - particle size reduction not possible due to nature of matrix.

12.2.4.2. Insufficient sample - less than the required 100 g minimum was available.

12.3. Review Requirements

12.3.1. Review all applicable holding times. If a holding time was exceeded, confirm that a holding time violation was properly documented in an NCM.

12.3.2. If Total analysis results are available, those results may be compared with the TCLP analysis results according to the following:

$$Total \geq 20 \times TCLP$$

Note: Assumes the sample is 100% Solids.

12.3.3. Total constituent analysis results can be used to demonstrate the TCLP protocol is unnecessary. In performing a TCLP analysis, there is a 20:1 dilution of the original sample with the leaching solution. Thus, if the “total constituent” result is less than 20 times the TC level, it is impossible for the leachate to “fail” and the TCLP does not need to be performed. For example, the TC level for lead is 5.0 mg/L (ppm). Therefore, if a sample of lead-contaminated soil contains less than 100 ppm total lead, a TCLP test need not be run to demonstrate that lead is less than the TCLP limit.

13. METHOD PERFORMANCE

13.1. Refer to individual analysis 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.

14. POLLUTION PREVENTION

14.1. This method does not contain any specific modifications that serve to minimize or prevent pollution.

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 Safety Manual for "Waste Management and Pollution Prevention."

15.2. The following waste streams are produced when this method is carried out.

15.2.1. Acidic waste from sample extract. This waste is collected in the laboratory in a designated container identified as "Acid Waste".

15.2.2. Buffer solutions. This waste can be poured down the drain with copious amounts of water.

15.2.3. Solid waste from sample extract, solid sample waste and used filter paper from the sample filtration step. This waste is disposed of in a designated container identified as "Solid Waste."

15.2.4. Flammable solvent waste and remaining TCLP extracts. This waste is disposed of in a flammable liquid solvent container identified as "Mixed Flammable Solvent Waste".

15.2.5. Glassware contaminated with acidic sample residue. Broken or unusable glassware is disposed of in a designated container identified as "Solid Waste." Glassware used in this test method is washed per the Glassware Washing SOP.

16. REFERENCES

16.1. Method 1311, Toxicity Characteristic Leaching Procedure, Revision 0, July 1992, SW-846 Final Update I.

16.2. Method 1312, Synthetic Precipitation Leaching Procedure, Revision 0, November 1994, SW-846 Update II.

16.3. Related Documents

16.3.1. Toxicity Characteristic: Corrections to Final Rule. Method 1311, Federal Register, Vol. 55, No. 126, Friday, June 29, 1990

16.3.2. Toxicity Characteristic: Final Rule. Method 1311, Federal Register, Vol. 55, No. 61, Thursday, March 29, 1990

16.3.3. Technical Background Document and Response to Comments, Method 1311, Toxicity Characteristic Leaching Procedure, USEPA/OSW, April 1989

- 16.3.4. SOP QA-003, Quality Control Program
- 16.3.5. SOP CORP-IP-0003NC: Acid Digestion of Aqueous Samples by SW846 and MCAWW 200 Series Methods
- 16.3.6. SOP NC-MT-012: Inductively Coupled Plasma-Atomic Emission Spectroscopy, Spectrometric Method for Trace Element Analysis, Method 6010B and Method 200.7
- 16.3.7. SOP CORP-MT-0005NC: Preparation and Analysis of Mercury in Aqueous Samples by Cold Vapor Atomic Absorption, SW-846 7470A and MCAWW 245.1
- 16.3.8. SOP CORP-MS-0002NC: Determination of Volatile Organics by GC/MS based on Methods 8260B
- 16.3.9. SOP CORP-MS-0001NC: GC/MS Analysis Based on Method 8270C
- 16.3.10. SOP NC-GC-038: Gas Chromatographic Analysis Based on Methods 8000B, 8021B, 8081A, 8082, 8151A, and 8015B
- 16.3.11. NC-OP-0031: Extraction Procedure for Chloriantaed Acid Herbicides based on Method 8151A
- 16.3.12. SOP NC-OP-032: Extraction and Cleanup of Organic Compounds from Waters and Soils, Based on SW846 3500 Series, 3600 Series, and 600 Series Methods
- 16.3.13. SOP NC-QA-0017: Standards and Reagents
- 16.3.14. SOP NC-WC-0010: pH Electrometric Method
- 16.3.15. SOP NC-QA-0014: Glassware Washing

17. MISCELLANEOUS

17.1. Modifications/Interpretations from Reference Methods

- 17.1.1. Section 11.2: Preliminary Evaluations. Section 7.1 of the source Method 1311 states that the sample aliquot used for the preliminary evaluation “...may not actually undergo TCLP extraction.” Section 7.1.5 of the source method indicates that the portion used for the preliminary evaluation may be used for either the ZHE or non-volatile extraction if the sample was 100% solid. Section 7.1.5 further indicates that if the sample was subjected to filtration (i.e., <

100% solid) that this aliquot may be used for the non-volatile extraction procedure only as long as sufficient sample is available (minimum 100 g). Samples which have been subjected to the oven-drying step may not be used for TCLP extraction because solid phase degradation may result upon heating.

- 17.1.2. Sections 11.3.7 and 11.4.21: Determination of Filtrate/Extraction Fluid Compatibility. Section 7.2.13 of the source method provides no guidance as to how to make this determination. As a result, the procedure herein was developed.
- 17.1.3. Section 9.2: TCLP Extraction Blanks. Section 8.1 of the source method states that a minimum of one blank for every 20 extractions “...that have been conducted in an extraction vessel.” TestAmerica has interpreted this to mean one blank per twenty samples leached per TYPE of leaching vessel (i.e., Bottle or ZHE) per leach fluid used.
- 17.1.4. Section 11.2.5.5.8.1: Determination of Appropriate Extraction Fluid. Method 1311 does not address the appropriate approach to take if the pH equals 5.0. This SOP requires that Fluid #1 must be used if the pH is less than or equal to 5.0.
- 17.1.5. Section 9.4: QA/QC - Matrix Spikes. Section 8.2 of the source method states “A matrix spike shall be performed for each waste type...” and “A minimum of one matrix spike must be analyzed for each analytical batch.” Further, Section 8.2.3 of the source method also states “The purpose of the matrix spike is to monitor the performance of the analytical methods used, and to determine whether matrix interferences exist.” The standard TestAmerica QAM is designed to address the performance monitoring of analytical methodology through the LCS program. A minimum of one MS and MSD will be prepared for each TCLP leachate batch. 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, the MS/MSD results have immediate bearing only on the specific sample spiked and not all samples in the batch.
- 17.1.6. Section 8.2.2 of the source method states that “In most cases, matrix spikes should be added at a concentration equivalent to the corresponding regulatory level.” The method also states “If the analyte concentration is less than one half the regulatory level, the spike concentration may be as low as one half of the analyte concentration but may not be less than five times the method detection limit”. For several analytes, spiking at the regulatory level is inappropriate to the range of analysis afforded by the determinative methods. Due to the wide range in these levels, TestAmerica spikes at the levels specified in the determinative SOPs.

APPENDIX A

TABLES

Table 3 - Toxicity Characteristic Analytes and Regulatory Levels (Final Rule)

Contaminant	mg/L
Arsenic	5.0
Barium	100.0
Benzene	0.5
Cadmium	1.0
Carbon tetrachloride	0.5
Chlordane	0.03
Chlorobenzene	100.0
Chloroform	6.0
Chromium	5.0
o-Cresols	200.0
m-Cresols	200.0
p-Cresols	200.0
Total Cresols (used if isomers not resolved)	200.0
2,4-D	10.0
1,4-Dichlorobenzene	7.5
1,2-Dichloroethane	0.5
2,4-Dinitrotoluene	0.13
1,1-Dichloroethylene	0.7
Endrin	0.02
Heptachlor (& epoxide)	0.008
Hexachlorobenzene	0.13
Hexachlorobutadiene	0.5
Hexachloroethane	3.0
Lead	5.0
Lindane	0.4
Mercury	0.2
Methoxychlor	10.0
Methyl ethyl ketone	200.0
Nitrobenzene	2.0
Pentachlorophenol	100.0
Pyridine	5.0
Selenium	1.0
Silver	5.0
Tetrachloroethylene	0.7
Toxaphene	0.5
Trichloroethylene	0.5
2,4,5-Trichlorophenol	400.0
2,4,6-Trichlorophenol	2.0
2,4,5-TP (Silvex)	1.0
Vinyl chloride	0.2

APPENDIX B

FIGURES

Figures 1 and 2 - Rotary Agitation Apparatus and Zero Headspace Extraction Vessel (ZHE)

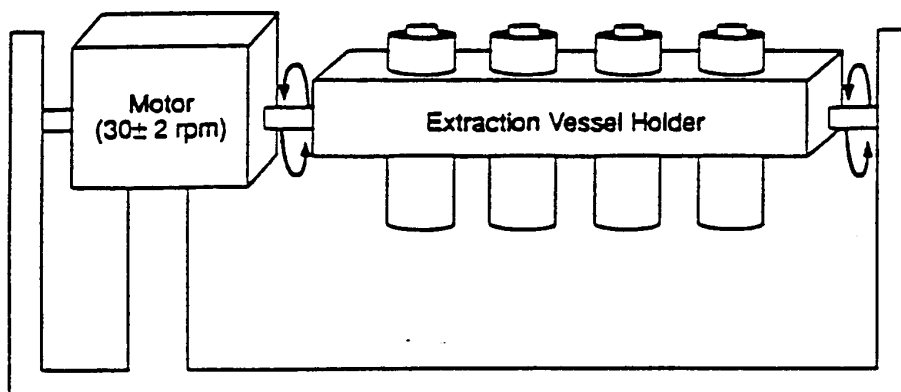


Figure 1. Rotary Agitation Apparatus

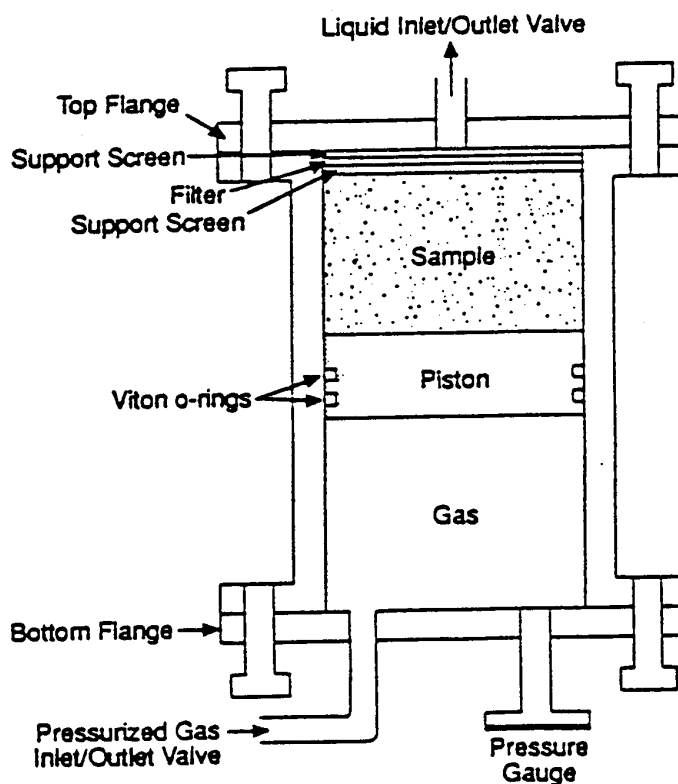


Figure 2 – Cross Section of Zero Headspace Extraction Vessel (ZHE)

Figure 3 - US Environmental Protection Agency Memorandum #35, Page 1



UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
WASHINGTON, D.C. 20460

OFFICE OF
SOLID WASTE AND EMERGENCY RESPONSE

MEMORANDUM # 35

DATE: June 12, 1992
SUBJECT: Notes on RCRA Methods and QA Activities
From: Gail Hansen, Chief *Gail Hansen*
Methods Section (OS-331)

This memo addresses the following topics:

- o 1992 Symposium on Waste Testing and Quality Assurance
- o SW-846 Update
 - Final Rule for January 23, 1989 Proposed Rule
 - Notice, Proposed Rulemaking for the Second Update to the Third Edition
- o Chlorofluorocarbon 113 (CFC-113) Solvent Replacement Update
- o Environmental Monitoring Methods Index (EMMI)
- o Sampling Work Group Formation
- o MICE Update
- o Oily Waste Analysis
- o Electronic SW-846 Availability.

Figure 3 - US Environmental Protection Agency Memorandum #35, Page 10 (cont'd)

Oil Waste Analysis

One of the most frequently asked questions on the MICE Service concerns the application of the TCLP, Method 1311, to oily wastes. Many callers request technical guidance on the extraction of oily wastes due to the difficulty in the filtration on these types of waste. In many cases, an oily waste does not filter completely due to premature clogging of the glass fiber filter. This can result in the retention of standing liquid on the glass fiber filter. Material that do not pass through the glass fiber filter at the conclusion of the filtration step is defined by the method as the solid phase of the waste. The solid phase is then subjected to the leaching procedure of the TCLP. For oily wastes, clogging of the glass fiber filter can result in an overestimation of the amount of solid material available for leaching.

To solve this problem, the Agency recommends a conservative approach, one that probably will overestimate the amount of leaching. Rather than performing the TCLP extraction on the unfiltered portion of the oily waste, assume the waste is 100% liquid (e.g., will pass through the glass fiber filter) and perform a totals analysis on the oily waste to determine if the oil exceeds the appropriate regulatory level.

Filterable waste oil generated during the TCLP must be analyzed for a variety of organic and inorganic analytes. The OSW recognizes the difficulty in achieving acceptable performance for the analysis of waste oil using methods currently provided in SW-846. As a result, the Agency will provide several new methods for the preparation and analysis of oil samples to the Organic Methods Workgroup in July. In addition, a microwave assisted digestion procedure should improve the analysis of metals and will be proposed as part of the Second Update of the Third Edition of SW-846. Brief descriptions of these techniques are provided below, for additional information on the organic procedures contact Barry Lesnik at (202) 260-7459. For additional information on microwave digestion contact Ollie Fordham (202) 260-4778.

The use of purge-and-trap (Method 5030) for volatiles in oil generally results in severe contamination of analytical instrumentation. Traps, transfer lines and chromatography columns may become contaminated with oil. This leads to elevated baselines, hydrocarbon background in subsequent analyses, and cross-contamination. Headspace (Method 3810) is currently allowed only as a screening procedure in SW-846. The Agency is evaluating the use of headspace in conjunction with isotope dilution mass spectrometry for the quantitative analysis of volatiles in oil. Headspace reduces interference problems encountered with purge-and-trap. However, headspace quantitation can be questionable because the distribution of analytes is not

APPENDIX C

LOGBOOK SHEETS

TESTAMERICA NORTH CANTON ZHE LOGBOOK

Pre- Prep Batch	Sample No.	Ext. Vessel ID	Matrix	Sample Wt (g)	Ext. Buffer ID	Date On	Time On	Analyst On	Date Off	Time Off	Initials Off	Tumbler ID	Tumbler Speed 30±2 RPM	Ext. Fluid ID	Initial ZHE Pressure	Final ZHE Pressure	Comments

TESTAMERICA NORTH CANTON ZHE LOGBOOK - MULTIPHASE

	A	B	C	D	E	F		G	H	
Sample Number	Weight of Container + Sample (g)	Weight of Container + Residue (g)	Difference (A – B) Sample Weight (g)	Weight of the Syringe (or vials) (g)	Weight of the Syringe (or vials) + Filtrate (g)	Difference (E – D) Filtrate Weight (g)	Volume of Filtrate* (mL)	Difference (C – F) Wet Solids Weight (g)	G x 20 Buffer Amount (mL)	Initials

* Filtrate volume only needed when filtrate is incompatible with leachate

TESTAMERICA NORTH CANTON TCLP LOGBOOK

Pre-Prep Batch	Sample No.	Parameters	Matrix	Pre-Test			% Dry Solids				Leach Start						Leach Finish				Tumbler ID	Comments
				Wt (g) Sample	pH after water	pH after heating	Wt (g) Sample	Wt (g) Filter	Filter + Particulates Wt (g)	% Dry Solids	Wt (g) Sample	Buffer # / ID	Date On	Time On	pH On	Analyst On	Date Off	Time Off	pH Off	Initials Off		

TESTAMERICA NORTH CANTON TCLP LOGBOOK – MULTIPHASE

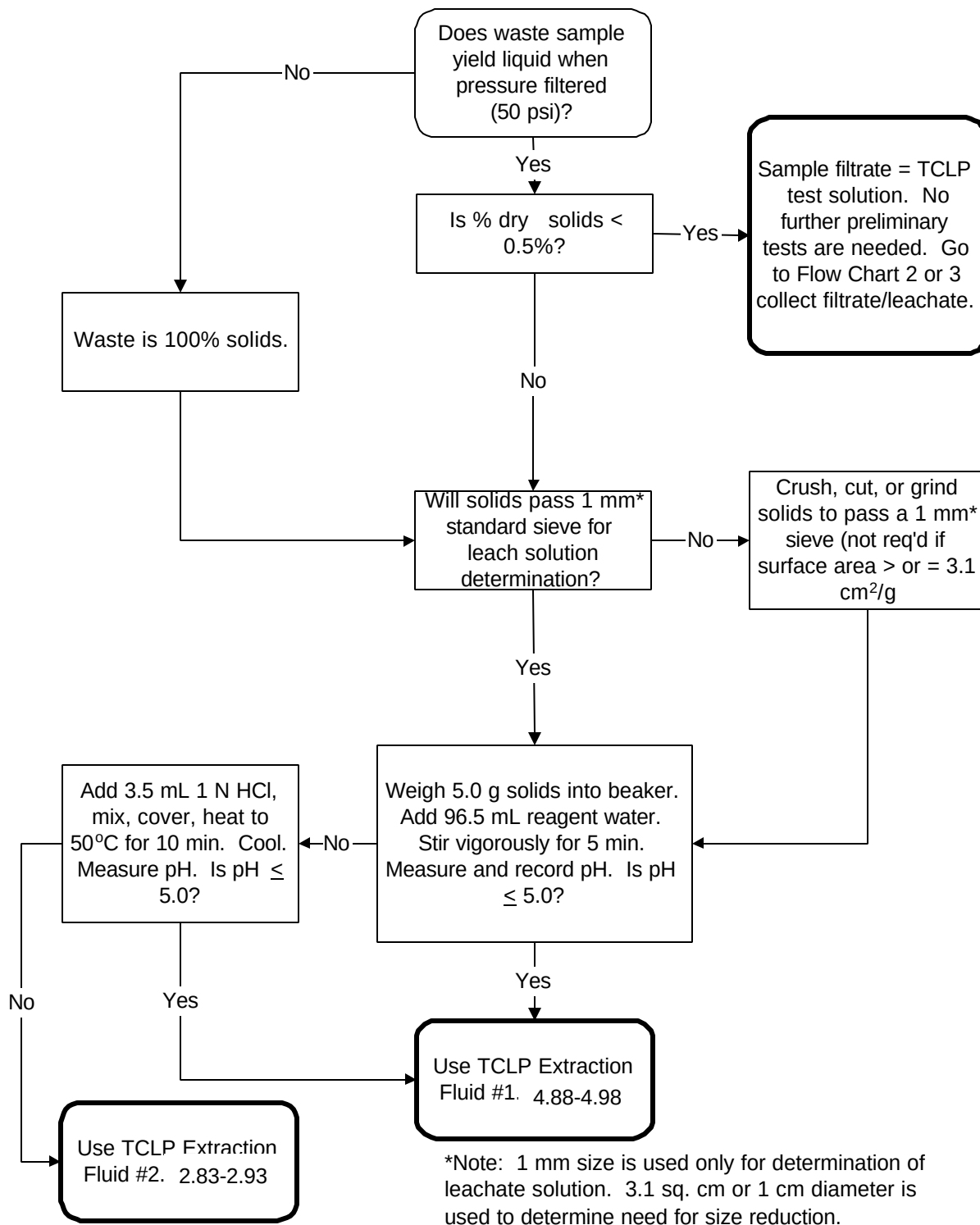
	A	B	C	D	E	F		G	H		
Sample Number	Weight of Container + Sample (g)	Weight of Container + Residue (g)	Difference (A – B) Sample Weight (g)	Weight of the Filtrate Bottle (g)	Weight of the Filtrate bottle + Filtrate (g)	Difference (E-D) Filtrate Weight (g)	Volume of Filtrate* (mL)	Difference (C-F) Wet Solids Weight (g)	G x 20 Buffer Amount (mL)	Initials	Comments

* Filtrate volume only needed when filtrate is incompatible with leachate

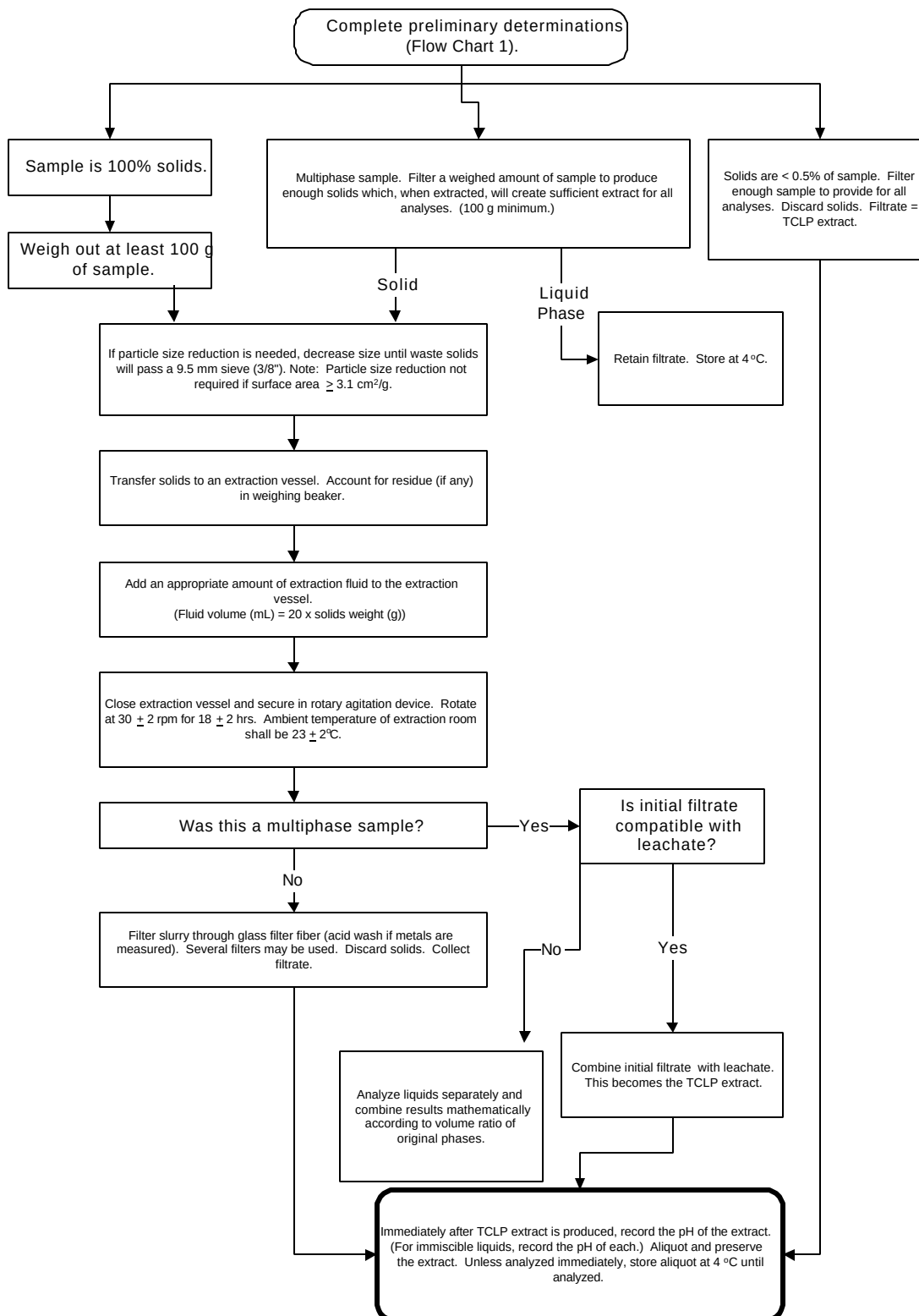
APPENDIX D

FLOW CHARTS

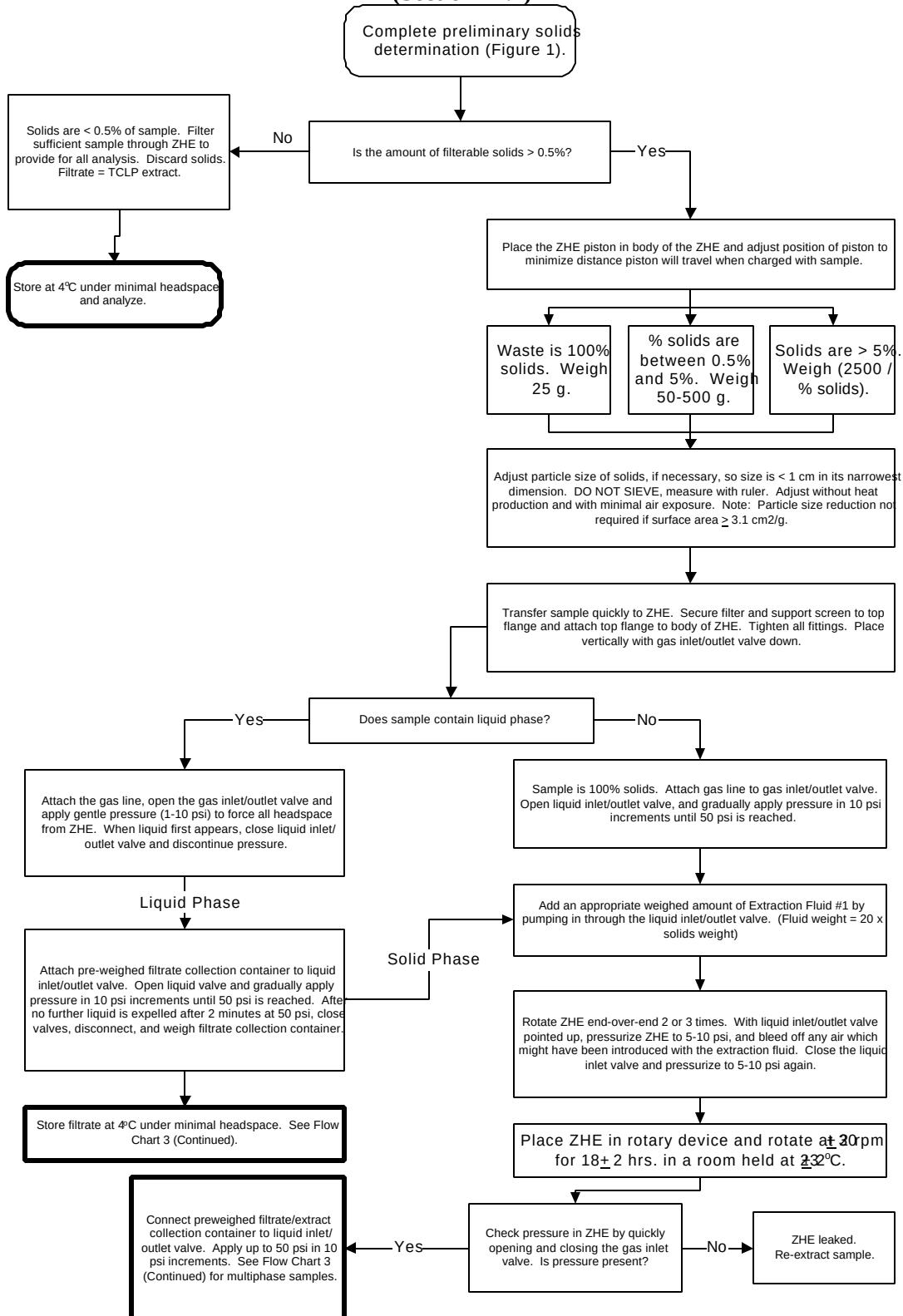
Flow Chart 1. Preliminary Sample Evaluation (Section 11.2)



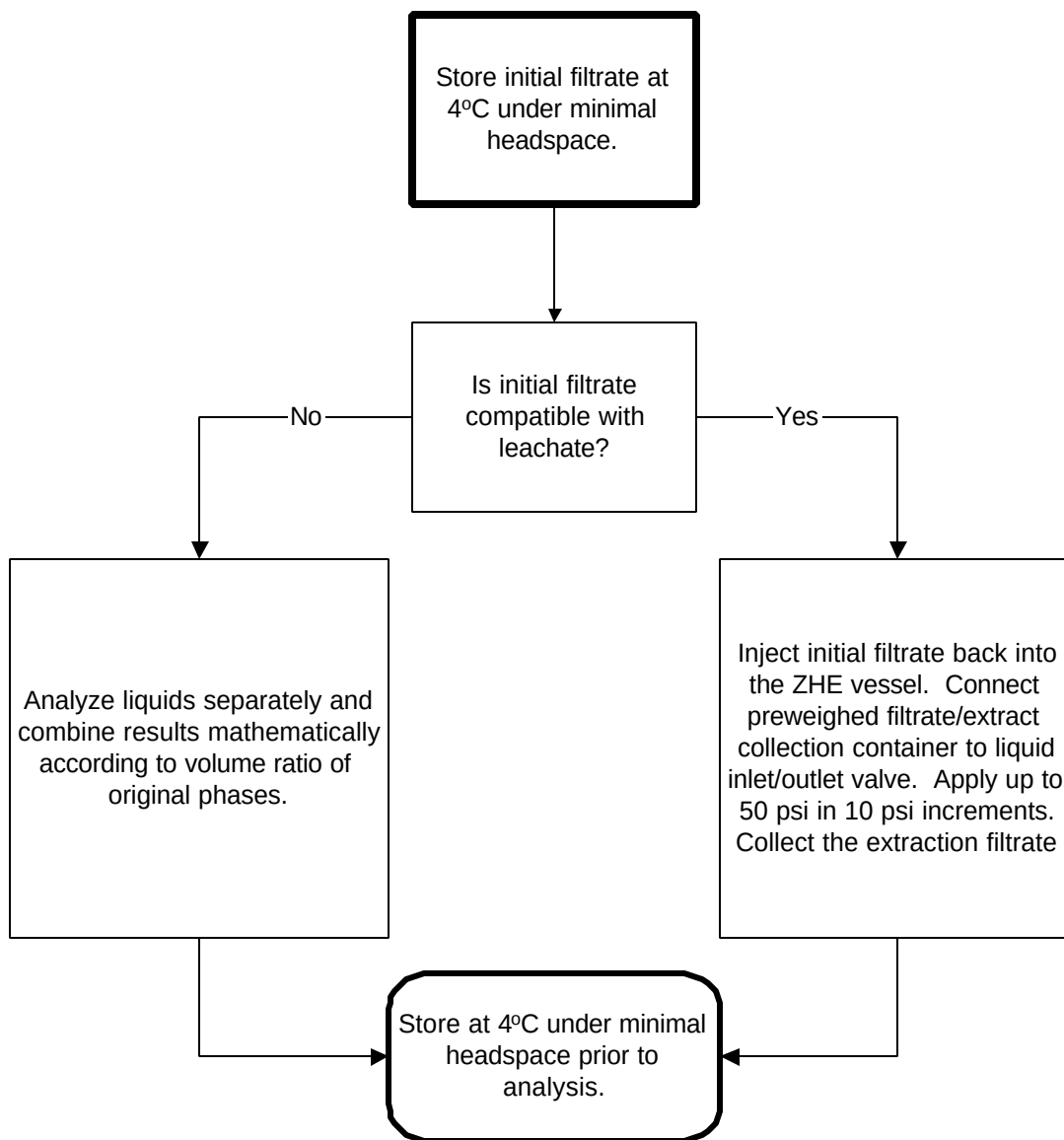
Flow Chart 2. Bottle Extraction, Non-Volatile Constituents (Section 11.3)



**Flow Chart 3. ZHE Extraction, Volatile Constituents
(Section 11.4)**

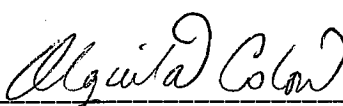
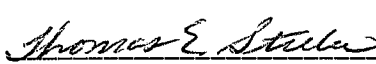
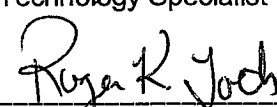
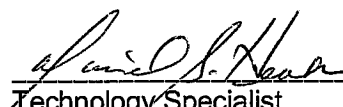
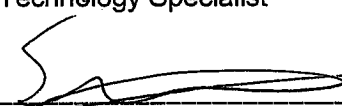


**Flow Chart 3. ZHE Extraction
(Continued)**



Title: GLASSWARE WASHING

[Method: None]

Approvals (Signature/Date):			
	4/17/08		4/23/08
Technology Specialist	Date	Technology Specialist	Date
	4-25-08		4/28/08
Technology Specialist	Date	Technology Specialist	Date
	4-23-08		4/18/08
Technology Specialist	Date	Health & Safety Coordinator	Date
	4/28/08		4/28/08
Quality Assurance Manager	Date	Laboratory Director	Date

This SOP was previously identified as SOP NC-QA-0014, Rev 6, dated 02/16/07

Facility Distribution No. _____

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TABLE OF CONTENTS

1. PURPOSE.....	4
2. RESPONSIBILITIES.....	4
3. SAFETY.....	4
4. PROCEDURES.....	5
5. DEFINITIONS.....	7
6. APPENDICES.....	8

1. PURPOSE

- 1.1. The procedure listed in this document will describe the standard operating procedures for washing glassware in the laboratories.
- 1.2. This document accurately reflects current standard operating procedures (SOP) as of the date above. All facility SOPs are maintained and updated as necessary by the laboratory.

2. RESPONSIBILITIES

- 2.1. It is the responsibility of the analyst to perform the analysis described herein in full compliance with this SOP.
- 2.2. It is the responsibility of the Laboratory Director, QA Manager, and departmental Supervisor of the facility to assure that the analysis described is performed in full compliance with this SOP. It is also their responsibility to supply adequate training, materials, and equipment to enable the analyst to perform this SOP correctly.

3. SAFETY

- 3.1. Employees must abide by the policies and procedures in the Corporate Safety Manual and this document.
- 3.2. 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 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	OSHA Exposure Limit (2)	Signs and symptoms of exposure/Unusual Hazards
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.

Material (1)	Hazards	OSHA Exposure Limit (2)	Signs and symptoms of exposure/Unusual Hazards
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.			

- 3.3. 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 when washing glassware. Disposable gloves that have been contaminated will be removed and discarded; other gloves will be cleaned immediately.
- 3.4. Exposure to chemicals must be maintained as low as reasonably achievable; therefore, unless they are known to be non-hazardous, all samples must be opened, transferred and prepared in a fume hood, or under other means of mechanical ventilation, where possible. Solvent and waste containers will be kept closed unless transfers are being made.
- 3.5. The glassware cleaning procedures that involve solvents such as methylene chloride will be conducted in a fume hood with the sash closed as far as the operation will permit.
- 3.6. 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 to a Laboratory Supervisor.
- 3.7. Glassware in contact with chemicals used in analytical procedures may be toxic or carcinogenic. Therefore, each piece of glassware should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals should be reduced to the lowest possible level.
- 3.8. Hands must not be placed in the glassware while washing. An appropriate scrub brush must be used to clean the inside of glassware. This will prevent the breakage of glassware while trying to force hands in or out of apparatus.

4. PROCEDURES

- 4.1. Any deviations from this procedure must be documented as a nonconformance with a cause and corrective action described.

4.2. Metals

- 4.2.1. Dirty glassware is taken to a central location and thoroughly rinsed. Any ink on the outside of the glassware is removed with acetone.
- 4.2.2. The glassware is immersed in a hot, soapy solution of water and laboratory detergent. An appropriate scrub brush or pad is used to scrub the glassware.
- 4.2.3. Rinse the glassware thoroughly three times with hot tap water and then rinse once with 1:1 nitric acid. Appropriate protective wear should be worn. If the glassware is still visibly dirty, or if spotting or beading occurs, repeat Section 4.2.2.
- 4.2.4. Rinse the glassware three times with analyte-free water, and place it in a clean drying area.
- 4.2.5. After air drying, the glassware should be spot- and stain-free. If not, then repeat the entire procedure. Seldom used items should be stored to minimize contamination.

4.3. Wet Chemistry

- 4.3.1. Dirty glassware is taken to a central location and thoroughly rinsed. Any ink on the outside of the glassware is removed with acetone.
- 4.3.2. The glassware is immersed in a hot, soapy solution of water and laboratory detergent. An appropriate scrub brush or pad is used to scrub the glassware. A mechanical, laboratory dishwasher equipped with a DI water rinse is also approved for glassware cleaning.
- 4.3.3. Rinse the glassware thoroughly three times with reagent water. If the glassware is still visibly dirty or if spotting or beading occurs, repeat Section 4.3.2.
- 4.3.4. After air drying, the glassware should be spot- and stain-free. If not, then repeat the entire procedure.
- 4.3.5. Place the glassware in a designated clean storage location free from interferences or contamination.

4.4. Semivolatile Organics

- 4.4.1. If high level contamination is suspected perform the appropriate high level cleaning before proceeding with the standard cleaning process described in Section 4.4.2 and following.
 - 4.4.1.1. Non-polar organics: if the glassware was most recently *wet* with a solvent, then rinse three times with that solvent, if *wet* with water, then rinse three times with acetone. Collect rinses in the appropriate solvent waste container. Allow the remaining solvent to evaporate from the glassware in a ventilation hood to reduce analyst exposure to the solvent.

- 4.4.2. After using a piece of glassware, it is thoroughly rinsed with hot tap water and carried to the dirty glassware area. If it is visibly dirty, it should be washed or soaked immediately rather than allow the residue to harden and become more difficult to wash.
- 4.4.3. The glassware is immersed in a hot, soapy solution of water and laboratory detergent (recommended pH >10). An appropriate scrub brush is used to scrub the glassware. Change the wash water (when it becomes cold, visibly dirty or is used on glassware with oil or sediment residue) by emptying, rinsing the inside of the sink with hot water and refill with hot water and detergent.
- 4.4.4. Rinse the glassware three times vigorously with hot tap water. If the glassware is still visibly dirty, or if spotting or beading occurs, rinse with 1:1 HCl and repeat Section 4.4.3.
- 4.4.5. After completing the hot tap water rinse, rinse the glassware three times with deionized water and place it in a clean drying area.
- 4.4.6. Place cleaned glassware used for semivolatiles in a muffle oven for approximately one hour at 400°C. Do not heat glassware with visible residue. Baked-on residue is much more difficult to remove. Do not heat volumetric glassware, including flasks, pipettes, syringes, etc., to avoid deformation.
- 4.4.7. Clean glassware is inverted (where applicable) and stored prior to use in a designated clean storage location.
- 4.4.8. Before use, glassware is to be pre-rinsed with the solvent inherent to a given procedure if stored more than one week.
- 4.5. Volatile Organics
 - 4.5.1. Rinse the glassware vigorously with hot tap water. If necessary, use a scrub brush to remove any remaining residue.
 - 4.5.2. Place the cleaned glassware in the drying oven until dry or overnight.
 - 4.5.3. Allow the cleaned and dry glassware to cool completely before use.
- 4.6. All Laboratory Groups
 - 4.6.1. All glassware washing brushes must be hung on hooks when not in use to prevent contamination and ensure proper drying.

5. DEFINITIONS

- 5.1. Refer to the glossary in the TestAmerica North Canton Quality Assurance Manual (QAM), current version.

6. POLLUTION PREVENTION

- 6.1. This method does not contain any specific modifications that serve to minimize or prevent pollution.

7. WASTE MANAGEMENT

- 7.1. 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 Safety Manual for "Waste Management and Pollution Prevention."

- 7.2. Waste Streams Produced by the Method

- 7.2.1. The following waste streams are produced when this method is carried out.

7.2.1.1. Flammable Solvent Rinse. Used solvents generated from glassware cleaning operations are placed in waste containers identified as "Mixed Flammable Solvent Waste"

7.2.1.2. Spent Acid Rinse. Concentrated used acids generated from cleaning glassware are collected in containers identified as "Acid Waste".

8. APPENDICES

- 8.1. See Table 1, Glassware Washing

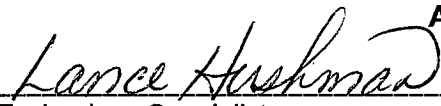
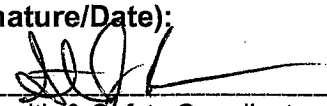
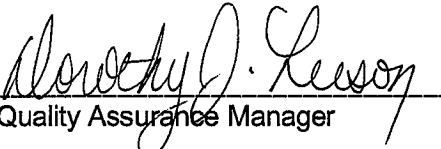
Table 1 Glassware Washing

	Metals	Organics	Wet Chemistry
Wash	Hot water, detergent solution	Hot water, detergent solution	Hot water, detergent solution
Rinse	3 times tap water 1 time 1:1 Nitric acid 3 times reagent water	3 times tap water 1 time 1:1 Hydrochloric acid* 3 times reagent water	3 times reagent water
Dry	Air	Muffle at 400°C for one hour	Air
Storage	Designated cabinets and shelves	Designated cabinets and shelves	Designated cabinets and shelves

*If the glassware is visibly dirty after washing in a hot detergent solution, the glassware is rinsed with 1:1 HCl and rewashed with a hot detergent solution.

Title: RECORDS INFORMATION MANAGEMENT

[Method: None]

Approvals (Signature/Date):			
	4-11-08		4-11-08
Technology Specialist	Date	Health & Safety Coordinator	Date
	4/4/08		4/14/08
Quality Assurance Manager	Date	Laboratory Director	Date

This SOP was previously identified as SOP No. NC-QA-019, Rev 6, dated 02/20/07

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TABLE OF CONTENTS

1. PURPOSE	3
2. RESPONSIBILITIES.....	3
3. SAFETY	3
4. PROCEDURES.....	3
5. DEFINITIONS.....	8
6. APPENDICES.....	10

1. PURPOSE

- 1.1. This document outlines the procedures associated with the storage and maintenance of all record created in the normal course of business such as laboratory data, client project files and all records generated in the course of laboratory operations.
- 1.2. This document accurately reflects current Standard Operating Procedures (SOP) as of the date above. All facility SOPs are maintained and updated as necessary.

2. RESPONSIBILITIES

- 2.1. It is the responsibility of the employee to perform the procedure described herein in full compliance with this SOP.
- 2.2. It is the responsibility of the Laboratory Director, QA Manager, and Department Supervisor of this facility to assure that the procedures described are performed in full compliance with this SOP. It is also their responsibility to supply adequate training, materials, and equipment to enable the employee to perform this SOP correctly.
- 2.3. Records are stored in a secure area at all times and can only be removed if charged out.

3. SAFETY

- 3.1. Normal office dependent safety precautions must be taken in performing this SOP. If personnel are required to perform any portion of the procedure in laboratory areas, appropriate personal protective equipment and precautions must be utilized.
- 3.2. Only 1.2 cubic foot boxes may be used to store all paper records due to risk of injury from larger sized boxes. These boxes have been found to weigh approximately 40 pounds on average.
- 3.3. When the forklift is used, individuals will have forklift training under OSHA standards. All safety equipment, such as a safety harness, will be used when operating the forklift.

4. PROCEDURES

- 4.1. Any deviations from this procedure must be documented as a nonconformance, with a cause and corrective action described.
- 4.2. Labeling Folders and Filing

- 4.2.1. Labels are printed in Central Reporting
- 4.2.2. The receptionist will align the client labels at the top score mark on the side-tab manila folders.
- 4.2.3. Folders are then given centralized reporting to be filed and initiate report and chromatograms. All reports and chromatograms are filed together to form a "Project File".
- 4.3. Records Transfer Process
 - 4.3.1. Record transfer forms are received in Record Management from all departments of the lab: Accounting, QA, EH&S, lab groups, etc. The reports/lab data is placed in a box by year, random order retention period, and a Record Transfer form is created for each box.
 - 4.3.2. The box is assigned an accession number (Cintas bar-coded label) and assigned a box location number. The date the reports are received are also written on the outside of the box, along with the client code range, Lab ID range and location. The label is stapled to the Record Transfer Form and both the accession number and box location is written on the form, copied and returned to the Records liaison. Then at scheduled times, the boxes that have been given labels are picked up and shelved.
 - 4.3.3. At no time will the Records Manager check to see if the information listed on the Record Transfer form is correct. It is the responsibility of the Records liaison to ensure the information is correct on the Record Transfer form.
- 4.4. Data Entry
 - 4.4.1. Open up Report Tracker, click on "Menu", and click on "New Batch".
 - 4.4.2. The next batch number will automatically appear.
 - 4.4.3. In the Customer Number Box, click on the down arrow; and select the appropriate customer code as it appears on the Record Transfer form.
 - 4.4.4. Entry date will automatically default to the current date.
 - 4.4.5. Click on the arrow, and the next screen will appear for adding a new ascension number.
 - 4.4.6. Click on "Add New Ascension"

- 4.4.7. In the Ascension Number field, enter the Andrews bar code number that was assigned to the Transfer form. Click “OK”.
- 4.4.8. Click on the down arrow in the Record Series column. Select and click. Series will appear in the box.
- 4.4.9. Enter the box location that was assigned on the Record Transfer form. Example: 30-D-2A.
- 4.4.10. In the Document Description box, enter the information that is located on the Record Title of the Record Transfer for (no more than 50 characters).
- 4.4.11. In the “Date Range From” field, enter the earliest month as it appears on the Record Transfer form. Example: January would be entered as 01/01/05.
- 4.4.12. In the “Date Range To” field, enter the latest month and last day of that month as it appears on the Record Transfer form. Example: January would be entered as 01/31/05.
- 4.4.13. Click on the “Review Date” field, delete the current date, enter the last month and day as the “Date Range To” field, enter the year as it appears on “Destruction Date” on the Record Transfer form. Example: If the month “From and To” were January, and the Destruction Date was a five-year retention, in the Review Date field, it would appear as 01/31/05.
- 4.4.14. If there are no details to add in the “Detail” field, click the arrow to next field and click back again. This will save all data entry. If you need to add another ascension number, repeat steps starting from Section 4.5.5.
- 4.4.15. If there are details to enter (such as Client Code, Lab I.D., etc.), after entering in the “Add New Ascension” field is complete, click the arrow at the bottom of the field. This will take you to the Details field.
- 4.4.16. Enter: Client Code, Lab I.D., Title, Case Number, SDG, if applicable. As it appears on the Record Transfer form. Once this is complete, click on arrow back to the “Add New Ascension” field.
- 4.4.17. If another ascension number is to be added, return to Section 4.5.5 and repeat.
- 4.4.18. After all data entry is finished in that batch, click arrows left and select “Print Batch”.
- 4.4.19. Print the batch out, and make two copies of each Record Transfer form. The original and one copy go to the originator of the form. The second copy and the Print Batch sheets are for the Records/Warehouse Manager’s records. The

Records Manager will Level I Review his copy with the Print Batch sheet, and make corrections as needed. He/she will then sign the "Print Batch" sheet as follows: "Level I Review, the date of review, and his/her initials". It will then be forwarded for Level II Review.

4.5. Editing a Batch

4.5.1. Open the Report Tracker. Click on "Edit Batch".

4.5.2. Enter the Batch Number. Click "OK". Click on the arrow, then click on the ascension number you wish to edit. Make the changes that are needed, and click arrow right to save changes. If changes need done in the "Detail" field, click arrow, make changes, and click arrows back left to save the changes.

4.6. Searching in the Report Tracker

4.6.1. Open Report Tracker. Click "Search". Enter details in the correct field, then click "Search".

4.6.2. Details can only be entered in one field at a time, except in the "Client Code" and "Lab I.D." fields. Multiple I.D. numbers and client codes can be entered here.

4.6.3. Once complete, in the "Search" mode, click "Back". This will return you back to the "Search" field.

4.6.4. Enter next item to search.

- Human Resource Records can be requested by: Human Resources and Directors
- Director Records can be requested by: Directors
- Medical Records can be requested by: Human Resources, Directors, and the Lab Nurse

4.7. Record Requests and Charge-Outs

4.7.1. There is a 30-day charge-out period for all records. The person who has charged out a record is responsible for that record until it is returned to the Records Department.

4.7.2. Records can be requested by stopping by, email, or calling the Records Department. A request for records must include the Lab ID, ascension number, or record title, including the year at a minimum. It is preferable to also include the client name and mail date.

- 4.7.3. Records that are stored off-site can be retrieved and delivered on Tuesday and Thursday. In an emergency, records can be retrieved from off-site storage within one hour at a cost of \$40.

4.8. Vital Records

4.8.1. SQL Server Backup Policy

- 4.8.1.1. The primary tool used for the backup of the SQL, Office and Target DB Servers will be Backup Exec for Windows NT (including the Agent for Microsoft SQL Server for NT). This will allow a singular backup format and strategy for the server's operating system, data files and databases.

- 4.8.1.2. The backups are scheduled as follows:

Scheduled Job	Job Type	Frequency	Retention Period	Storage Location
Monthly	Full (including OS, data, and databases) – SQL and Office servers only	First Monday of Month 1:00 AM	1 Year	On-site Fire Safe
Weekly	Full (including OS, data, and databases) – SQL and Office servers only	Every Monday 1:00 AM	4 Weeks	On-site Fire Safe
Weekly	Full (including OS, data, and databases) – Target DB servers only	Every Monday 1:00 AM	Indefinitely	On-site Fire Safe
Daily	Differential (OS and data) Full (including OS, data and databases) – Target DB only Full (including OS, data, and databases) – Target DB servers only	Tuesday-Friday 1:00 AM	1 Week	On-site Fire Safe

- 4.8.1.3. The backups should be configured as follows:

- 4.8.1.4. SQL, Office Servers and Target DB

- 4.8.1.5. Server Backup Media Set Should have Overwrite Period = 7 day, Append Period = Infinite
- 4.8.1.6. Job Properties
 - 4.8.1.6.1. Append to media; overwrite if no appendable media is available.
 - 4.8.1.6.2. Device – Quantum 0
 - 4.8.1.6.3. Media Set – Server Backups
 - 4.8.1.6.4. Write Checksums to Media (True)
 - 4.8.1.6.5. Compression – Hardware (if available, otherwise software)
 - 4.8.1.6.6. SQL – DATABASE – Backup Entire Database
- 4.8.1.7. Backup Method for Files
 - 4.8.1.7.1. Full Backup – NORMAL – Back Up Files – Reset Archive Bit
 - 4.8.1.7.2. Differential Backups – DIFFERENTIAL – Change Files
- 4.8.1.8. Backup Selections
 - 4.8.1.8.1. Include all data directories
 - 4.8.1.8.2. SQL Server Databases include only non-QuantIMS objects of DATAMIRROR and include all other databases except PUBS and NORTHWIND.
- 4.8.1.9. The monthly backup tape consists of the first Monday of the month of the weekly backup tapes. There is no additional monthly backup performed.
- 4.8.1.10. All backups should be monitored and verified by the LAN Administrator. Execution of the backups, and tape handling is also the lab LAN Administrator's responsibility.
- 4.8.1.11. Once the tapes are brought to Records, they are logged in on an SQL Backup Tapes logsheet, and then returned to be recycled.

4.8.2. Blueprints

- 4.8.2.1. The building blueprints have been duplicated on aperture cards, which are stored off-site, in a vault at Cintas Records Management. A copy is kept on-site in a fireproof cabinet.

4.9. Record Retention

- 4.9.1. Records are retained for a total of five years unless a client specifies other retention requirements. Drinking water analyses are kept ten years, and NELAP requires five years
- 4.9.2. In the event that the laboratory transfers ownership or goes out of business, the records will be maintained until the scheduled retention period has been met. Clients with contractual agreements for the return of their records will be contacted. Those records will be returned at the client's expense. Proof of ownership and responsibility will be required before the records may be released.
- 4.9.3. This procedure ensures compliance with the TestAmerica Record Retention Policy, P-L-001.

4.9.3.1. Special Record Retention Requirements

Program	Retention Requirement
Ohio – Drinking Water	10 years
Michigan Department of Environmental Quality – all environmental data	10 years
Navy Facilities Engineering Service Center (NFESC)	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

- 4.9.4. At the end of the specified retention period, records are destroyed per criteria in SOP NC-QA-013, Inventory/Warehouse Control.

5. DEFINITIONS

- 5.1. Refer to the glossary in the TestAmerica North Canton Quality Assurance Manual (QAM), current version.
- 5.2. Chromatogram(s) refers to raw data generated from laboratory organic analyses.
- 5.3. Field - one part of record that contains specific information. (Example: the box number would be one field)
- 5.4. Record - consists of a number of information fields. (Example: the box number, location and department would make up a record)
- 5.5. Batch - one or more boxes entered for a single department.
- 5.6. Accession number - six digit bar code number used to identify a single box.
- 5.7. File boxes - the standard box used is 1.2 cubic feet in size and weighs approximately 35 - 40 pounds full.
- 5.8. Vital records - records that are essential to protecting the assets of the company and its ability to continue operations are considered vital.
- 5.9. References
 - 5.9.1. CW-Q-S-001, Corporate Document Control and Archiving, current version
 - 5.9.2. P-L-001, Record Retention, current version
 - 5.9.3. NC-QA-013, Inventory/Warehouse Control, current version

6. APPENDICES

- 6.1. Example Records Transfer Form
- 6.2. Department Codes for Report Tracker

Appendix 1: Example Records Transfer Form

RECORD TRANSFER FORM

Directions: 1) Complete one form per box and detail contents as much as practical. 2) Write record title on side of the box with a handle and year of content. 3) Send Record Transfer Form approved and signed by Department Record Liaison to records. 5) After receiving barcode label attached under the box handle. 6) The location, which is to be written on the right side of box, (same side with handle) and circled. 7) Place Record Transfer Form in box. 8) Place box in centralized record pick-up area.

NAME:

DATE: (RELEASED BY)

Record Series Number: 0069 – QUAL ASSUR

Department Code: 00QA – Quality Assurance

Record Title:

(No more than 50 Characters) (Example: 2000 Sample Removal Requests, January 2000 Batch QC, etc.)

Date of Records:

Retention Period:

Destruction Date:

(One year per box ONLY)

(Refer to Retention Policy, LEG-004)

(Year Only)

Matrix: Paper

Film/Fiche

Diskette

Mag Tape

CD

Other

Record Liaison:

Accession Number:

Box Location:

Records Use Only:

Batch#: _____ **AS400 entry:** _____ **Report Tracker entry:** _____

Contents: (Be very specific. List when applicable: client code, lot number, name of client, batch numbers, instrument number, month date range, etc.)

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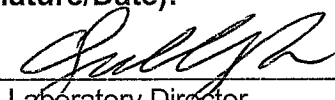
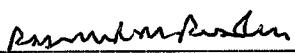
Rev 2, 02/22/07 jse

Appendix 2: Department Codes for Report Tracker

Code	Department	Code	Department
00HR	Human Resources	0MSS	GC/MS Semivolatiles
00IS	Information Services/Technology	0MSV	GC/MS Volatiles
00PM	Project Management	0WET	Wet Chemistry
00QA	Quality Assurance	MAIN	Maintenance
0ADM	Administration	OPER	Operations
0EHS	Environmental Health & Safety	PURC	Purchasing
0EXT	Organic Extractions	RMGT	Records Management
0FAS	Field Analytical Services	SALE	Sales
0GCS	GC Semivolatiles	SAMC	Sample Control
0GCV	GC Volatiles	SHIP	Shipping
0LAW	Legal	TRAI	Training
0MET	Metals		

**Title: EVALUATION OF METHOD DETECTION LIMITS
FOR CHEMICAL TESTS**

[Method: None]

Approvals (Signature/Date):			
	04/29/08		4/29/08
Quality Assurance Manager	Date	Laboratory Director	Date
	4-30-08		
Technical Director	Date		

This SOP was previously identified as SOP No. NC-QA-0021, Rev 5, dated 04/25/07

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TABLE OF CONTENTS

1. <i>Purpose</i>	3
2. <i>Responsibilities</i>	3
3. <i>Safety</i>	3
4. <i>Procedures</i>	3
5. <i>Definitions</i>	9
6. <i>Data Analysis and Calculations</i>	9
7. <i>References</i>	14
<i>Appendix</i>	15

1. PURPOSE

- 1.1. The document describes the policies and procedures relative to the evaluation, acceptance and iterative process of method detection limits for chemical tests. This SOP is based on TestAmerica Policy CA-Q-S-006 (Method Detection Limit Studies) and 40 CFR Part 136 (Appendix B).
- 1.2. This document accurately reflects current standard operating procedures (SOP) as of the date above. All facility SOPs are maintained and updated as necessary by the laboratory.

2. RESPONSIBILITIES

- 2.1. It is the responsibility of the employee to perform the procedure described herein in full compliance with this SOP.
- 2.2. It is the responsibility of the Laboratory Director, QA Manager, and departmental Supervisor of this facility to assure that the policy and analyses described are performed in full compliance with this SOP and the corresponding analytical SOPs. It is also their responsibility to supply adequate training, materials, and equipment to enable the employee to perform this SOP correctly.

3. SAFETY

- 3.1. Normal office dependent safety precautions must be taken in performing this SOP. If personnel are required to perform any portion of the procedure in laboratory areas, appropriate personal protective equipment and precautions must be utilized.

4. PROCEDURES

- 4.1. Any deviations from this procedure must be documented as a nonconformance, with a cause and corrective action described.
- 4.2. Method Detection Limit Study (MDL Study) Evaluation
 - 4.2.1. The spike concentration and the final calculated concentration for each replicate of an MDL Study is entered into an Excel[®] spreadsheet. The Excel spreadsheet calculates the

average, the average percent recovery, the precision and the MDL based on the seven replicate analyses. An example spreadsheet is included in Section 6 of this SOP.

- 4.2.2. MDLs are representative of the performance of the method in the laboratory and can be influenced by the performance of individual instruments. Therefore, one MDL per matrix performed on each instrument may be determined.
- 4.2.3. Appendix B of 40 CFR Part 136 requires the use of 7 replicates. If more than 7 replicates are used for MDL Studies, data from all replicates must be used in calculating the MDL. In these cases, the appropriate student's t-value (n-1 degrees of freedom, 99% confidence level) must be used in the calculation of the MDL. (See 40 CFR Part 136, Appendix B). MDLs may be determined on a matrix, method, and instrument specific basis.

4.3. MDL Study Acceptance Criteria

- 4.3.1. See 40 CFR Part 136, Appendix B for MDL acceptance criteria.
- 4.3.2. Detection Limit studies are also evaluated against any associated IDL evaluations, previous MDL studies, established RLs, any method specified IDLs or MDLs, and any other criteria in TestAmerica North Canton Quality Assurance Manual (QAM), Supplemental Practices for DoD Project Work SOP (NC-QA-0016), and MDL Policy (CA-Q-S-006).
- 4.3.3. All studies must be reviewed by appropriate QA personnel and laboratory management staff prior to data release.
- 4.3.4. DoD program requirements are noted in the "Corporate Appendix B" attachment of this SOP.

4.4. Security and Validation

- 4.4.1. All MDL spreadsheets are forwarded to the QA department for final review and centralized filing.
- 4.4.2. The MDL spreadsheets are placed on the public QA drive under MDL and separated by year after final review and sign-off by QA personnel.
- 4.4.3. The QA drive is a "read only" drive and only designated personnel can alter data residing there.

4.5. Spreadsheet Instructions

- 4.5.1. Before using the application the public directory must be set to drive N:\ in the computer used for data entry. This is absolutely critical to ensure that the file will automatically save to the right place.
- 4.5.2. The following directory structure must be in the public drive. N:\QA\MDLSTUDY.
- 4.5.3. The file CANMDL.xls must be saved under the N:\QA directory, and must be write-protected.
- 4.6. Using the application
 - 4.6.1. Open Excel.
 - 4.6.2. Retrieve the following file... N:\QA\CANMDL.XLS.
 - 4.6.3. The first screen that will appear is shown in Figure 1.

Figure 1. Opening screen of MDL application

- 4.6.4. Note that the opening screen has several buttons that define the basic functionality of the application. Whenever this application is used, all of the spreadsheet functionality (saving files, printing reports, etc.) is handled using these buttons. Since using menuubar items can cause unexpected results, many of them have been turned off. More specifically...
 - 4.6.4.1. The **Save File** button is used to save the file in a specified format to a specified directory. The menuubar functionality (File Save and File Save As) has been inactivated.

4.6.4.2. The **Print Report** button is used to print the MDL report in a specified format. The menubar functionality (File Print) has been inactivated.

4.6.4.3. The **Exit Excel** button is used to exit the program. The menubar functionality (File Exit) has been inactivated. Although the user can still exit Excel using the control button at the top left corner of the application window, this practice is strongly discouraged. Doing so will create difficulties the next time Excel is used.

4.6.5. Press the **Fill header** button.

4.6.5.1. A series of four dialog boxes like the one Figure 2 will request some information about sample prep and analysis dates, instrument ID, and analyst name. Enter the requested information and either press the OK button, or press the return key after each one.

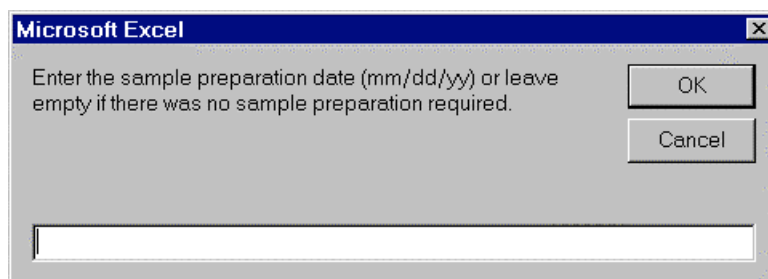


Figure 2. Fill header sample screen

4.6.5.2. After the four boxes, a dialog box like the one in Figure 3 will appear in which you will make selections for other information that is needed in the documentation. Select one of the choices for each of the pieces of information requested... to view the available options, press the down arrow, move to the right choice and click on it.

4.6.5.3. If none of the choices presented describes the MDL study (or any portion of the header), notify the QA department for modification. However, you can still proceed. Select the first option, which is normally blank, and when the application becomes active again, type in the correct information. The one piece of information that must be selected in this dialog box is a method. The reason for this is that the method selected drives the automatically assigned filename.

MDL Calculation Header Setup

To ensure consistency between the information provided with MDL study data, select the a value for each entry by selecting the correct choice from the drop-down box. To view

Select a lab location: Canton

Select a production group:

Concentration units:

Methods

Sample preparation method:

Analysis method:

Other applicable analysis methods:

Matrix: Matrix material used:

OK Cancel

Figure 3. Dialog box in the fill header functionality

4.6.6. After the header information is filled, the MDL data is entered. There are several options.

4.6.6.1. For data which is not contained in data files, or for data that involves one or two parameters, type the MDL data manually. The first column is the name of the parameter, followed by spike amount (in the units reflected in the header) and MDL replicate concentrations found (in the units reflected in the header).

4.6.6.2. Import of electronic data from the following applications is supported: Target (GC/MS VOA, GCVOA, GCMS SemiVOA, and GC SemiVOA only)

4.6.6.2.1. For Target data import, the user must convert the mdl.rp files from the Target file server to .csv (comma separated value) files. There will be one mdl.rp file for each MDL study.

4.6.6.2.2. Go to Network Neighborhood to find the batch file name specified on the Target MDL report. For example
[\\qcanoh04\dd\chem\GCS\2hp4.i\040401-0.b.](#)

4.6.6.2.3. Open the .csv file.

4.6.6.2.4. Save the file as an Excel® Spreadsheet in the N:\QA folder. Close the instrument file in Network Neighborhood.

- 4.6.6.2.5. Save the final version of the spreadsheet in N:\QA.
- 4.6.6.3. Open the following: **N:\QA\CANMDL.xls** (A prompt will appear concerning macros, choose Enable Macros) The Spreadsheet will open.
- 4.6.6.4. Choose the button labeled Header and fill in the information as prompts appear.
- 4.6.6.5. Open the spreadsheet saved in Section 4.6.6.2.11.
- 4.6.6.6. Highlight all the compound names. Select Copy from the Edit menu. Go to the Window Menu and select the MDL spreadsheet. Place the cursor in the first space under Compound and Select PASTE from the Edit menu. The entire list of compound names will be copied into the MDL spreadsheet. Repeat the process with the replicate results.
- 4.6.6.7. Close the revised replicate spreadsheet..
- 4.6.6.8. On the MDL spreadsheet, enter the spike concentration.
- 4.6.6.9. Select the CALCULATE MDL button. The MDL will be calculated in a few seconds. Select VIEW MDL to verify this. The results screen (Figure 4) has precision and accuracy data as well as the MDL. While there are no specified criteria for precision or accuracy, the replicate values should be compared to the spike amount to ensure a reasonable recovery for the analyte or test. The ratio of the spike to the MDL must be between 1 and 10 for 40 CFR criteria. For DoD criteria, the ratio must be 1:5 for waters and 1:10 for solids. If this criteria is not met, the study is invalid and needs to be repeated.
- 4.6.6.10. If any replicate value is not detected, the MDL study is invalid and must be repeated. The only exception to this rule would be if the replicate was invalid for a known cause (example: did not inject, did not spike correctly, etc.). The replicate may then be replaced with documentation as to the reason for replacement.

Microsoft Excel - QK99A28.XLS

Return to Raw Data

Lab Name:	Quanterra Canton	Applicable matrix:	Soil
Group:	GC/MS VOA	Matrix used:	Ottawa sand
Prep. Method:	Purge and trap	Conc. Units:	ug/Kg
Analysis Method:	SW846 8260B	Prep. Date:	12/29/08
Other applicable methods:	SW846 8260A	Analysis Date:	12/29/08
Standard Analysis Code (SAC):	OK	Instrument ID:	50-2
Filename:	QK99A28	Analyst:	Richard Quayle

Compound/parameter	Spike	Avg	Range		S.D.	%RSD	MDL	Ratio	n	Comment
			%Rec	%Rec						
Dichlorodifluoromethane	250	97.4	81.7	113	13.100	5.37	41	6.1	7	
Chloromethane	250	94.6	85	104	8.010	3.38	25	9.9	7	
Bromomethane	250	122	105	140	14.500	4.74	46	5.5	7	
Chloroethane	250	142	105	178	30.400	8.58	36	2.6	7	
Carbon Disulfide	250	98	85.5	111	10.500	4.27	33	7.6	7	
Acrylonitrile	2500	82.3	57.5	107	206.000	10	650	3.9	7	
Vinyl acetate	250	53	31.4	74.6	18.000	13.6	57	4.4	7	
2-Chloroethyl vinyl et	500	87.3	55.2	119	53.400	12.2	170	3.0	7	
4-Methyl-2-pentanone	250	88	50.8	125	31.000	14.1	97	2.6	7	
2-Hexanone	250	93.3	16.4	170	64.100	27.5	200	1.2	7	
1,1,2-Trichloroethane	250	97.5	82.1	113	12.800	5.26	40	6.2	7	
Dibromochloromethane	250	87	76.4	97.7	8.880	4.08	28	9.0	7	
Bromoform	250	82.5	72.1	92.9	8.650	4.19	27	9.2	7	
1,1,2,2-Tetrachloroeth	250	77.6	63.9	91.3	11.400	5.87	36	7.0	7	
Freon-113	250	124	112	135	9.280	3	29	8.6	7	
Acetonitrile	2500	71.8	24.4	119	395.000	22	1200	2.0	7	
1,4-Dioxane	12500	78.7	48.8	109	#####	12.7	3900	3.2	7	

Figure 4. MDL study results screen

4.6.6.11. Press the **Save File** button. A filename has been automatically assigned to the spreadsheet based on the method code and analysis date. It will save to the **N:\QA\MDLSTUDY** directory with the assigned filename. At this time the file can be renamed, for example, UX10_25mL purge water_ (date).

4.6.6.12. Press the **Print Report** button. The MDL study report has three parts: (1) a cover sheet with a review checklist and other information, (2) the MDL values, and (3) the data that you entered. Complete the laboratory portion of the review checklist in the cover page, and forward to QA.

5. DEFINITIONS

- 5.1. Refer to the glossary in the TestAmerica North Canton Quality Assurance Manual (QAM), current version

6. DATA ANALYSIS AND CALCULATIONS

- 6.1. MDL study report

- 6.1.1. The MDL study report consists of three parts: (1) The MDL study checklist, which is used to document that the report was reviewed and that the results were entered into QuantIMS; (2) The MDL results, which has the MDL value (calculated to two significant figures), the spike amount, the average recovery and the recovery range (low and high values), and the spike-to-MDL ratio; and, (3) The MDL raw data (the values obtained from analysis of the replicate spikes used to calculate the MDL).
 - 6.1.2. Each page of the MDL report has the same header, which includes information about the method used, the matrix used, the concentration units, dates, instrument and analyst.
- 6.2. Data review
- 6.2.1. Technical review
 - 6.2.1.1. The Group Leader or designee reviews the MDL values. In some cases (especially inorganic tests), other considerations affect the laboratory's ability to use the calculated MDL as a measure of sensitivity. In such cases, the Group Leader may write under the *Comments* column the most reasonable MDL value. Keep in mind that this value may not be less than the calculated MDL.
 - 6.2.1.2. In Part 1 of the MDL study report, the Group Leader or designee documents whether the method was changed to perform the MDL study (as compared to routine sample analysis), whether a method blank was processed with the samples, whether the analysis was performed over several days, and whether any data was rejected. With the exception of the question about a method blank, any "Yes" answer requires an explanation. The Group Leader or designee should also note whether ratios were met, and whether the RL is less than the MDL.
 - 6.2.1.3. The QA Department reviews the MDL results in a second level review. The QA Department reviews and documents whether the appropriate matrix was used, whether the ratios were between 1 and 10, if the MDLs are less than the standard reporting limits. If any spike-to-MDL ratio exceeds 10, corrective action may be required, including repeating the MDL study for the affected analytes.
 - 6.2.1.4. The QA Department is notified when the Group Leader or designee review is complete, and final studies are complete.
 - 6.2.1.5. Additional Requirements for MDLs are listed in SOP NC-QA-0016 and CA-Q-S-006.

6.2.2. MDLs may be instrument specific. MDL data meeting ratio and reporting limit criteria from all instruments is compiled into a separate master spreadsheet for each matrix. The spreadsheet is printed for review by the QA staff. See Figure 5.

[illegible]

Figure 5: Master Spreadsheet per method and matrix

6.2.3. Once the spreadsheet has been reviewed and approved by the Group Leader and QA, the updated reference data is entered into QuantIMS via the DLMS (Detection Limit Management System) program as described in the following steps.

6.2.3.1. A structured analysis code is assigned for each combination of method, preparation, and QC requirements. This code is required to access the information in DLMS. Each method code can have several combinations of SACs and these can be chosen at one time in DLMS. See Figure 6.

BAC Select

Available SAC's				
Matrix	Prep	Method	QC	Location
I	- 25	- QK	- 01	- 01
I	- 25	- QK	- 2J	- 01
I	- 25	- QK	- 2V	- 01
I	- 25	- QK	- 3S	- 01
I	- 25	- QK	- 5B	- 01

Details

Location: 01 - Custians - North Canton

Matrix:

Method: 0K - Volatile Organics, GC/MS (B210B)

Prep:

QC:

OK Close

Add Edit View Delete Version Help

Figure 6. SAC listing in DLMS

- 6.2.3.2. Select one of the SACs listed. A screen similar to Figure 7 will appear. Enter the new MDL values and analysis dates for each analyte or test to be updated. Press Update when finished. The screen is now the same as in Figure 6.
- 6.2.3.3. The new MDL values can now be copied to all other SACs as needed, keeping in mind client specific requirements. Choose the SAC with the new MDL values, press Copy. A screen similar to Figure 6 will appear. Choose from the selections and press OK. Select the new SAC from the list and press enter. A screen similar to Figure 7 will appear. Press enter and the MDL values will be copied into the new SAC.

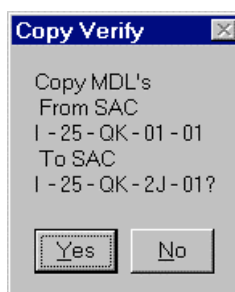


Figure 7: Copy function in DLMS

- 6.2.3.4. When all SAC combinations have been updated, close the DLMS program.
- 6.2.4. Verification of the newly entered MDLs will be available to the lab on the next day via the QC Browser program.
 - 6.2.4.1. To check data entry, open the QC Browser program
 - 6.2.4.2. Enter the SAC components and select “All Analytes” and Load. Analytes will appear for the SAC entered
 - 6.2.4.3. Select “Date Options.” Set the date to the activation date of the newly entered MDLs.
 - 6.2.4.4. Select the Report function. Choose the MDL and RL version of the report.
 - 6.2.4.5. Print the report and compare to the highlighted spreadsheet to verify the correct MDL values were entered into LIMS.

QuickReport version 1.1

Page 1 of 3

STL Detection Limit Summary

Structure Name: 111-000-01-01
Target Analyte: 111-000-01-01
Method: 111-000-01-01-01
Customer: 111-000-01-01-01

MO: 111-000-01-01
QC: 111-000-01-01-01
Lab: 111-000-01-01-01

Item	Compound	Unit	MDL	MDL	MDL
11	Acetone	10	10%	0.1	10%
22	Acetone	20	10%	1.5	10%
33	Acetone	30	10%	1.2	10%
44	Acetone	40	10%	1.6	10%
55	Acetone	50	10%	0.22	10%
66	Acetone	60	10%	0.29	10%
77	Acetone	70	10%	0.24	10%
88	Acetone	80	10%	0.14	10%
99	Acetone	90	10%	0.19	10%
100	Acetone	100	10%	0.11	10%
111	Acetone	110	10%	0.36	10%
122	Acetone	120	10%	0.26	10%
133	Acetone	130	10%	1.2	10%
144	Acetone	140	10%	0.39	10%
155	Acetone	150	10%	0.29	10%
166	Acetone	160	10%	0.29	10%
177	Acetone	170	10%	1.2	10%
188	Acetone	180	10%	2.1	10%
199	Acetone	190	10%	0.2	10%
200	Acetone	200	10%	0.16	10%
211	Acetone	210	10%	0.19	10%
222	Acetone	220	10%	0.25	10%
233	Acetone	230	10%	0.19	10%
244	Acetone	240	10%	0.2	10%
255	Acetone	250	10%	0.2	10%
266	Acetone	260	10%	0.2	10%
277	Acetone	270	10%	0.2	10%
288	Acetone	280	10%	0.2	10%
299	Acetone	290	10%	0.2	10%
300	Acetone	300	10%	0.2	10%
311	Acetone	310	10%	0.22	10%
322	Acetone	320	10%	0.26	10%
333	Acetone	330	10%	0.14	10%
344	Acetone	340	10%	0.14	10%
355	Acetone	350	10%	0.21	10%
366	Acetone	360	10%	0.21	10%
377	Acetone	370	10%	0.19	10%
388	Acetone	380	10%	0.17	10%
399	Acetone	390	10%	0.12	10%
400	Acetone	400	10%	3.3	10%
411	Acetone	410	10%	0.28	10%
422	Acetone	420	10%	0.39	10%
433	Acetone	430	10%	0.24	10%
444	Acetone	440	10%	0.24	10%
455	Acetone	450	10%	0.21	10%
466	Acetone	460	10%	0.21	10%
477	Acetone	470	10%	0.2	10%
488	Acetone	480	10%	0.2	10%
499	Acetone	490	10%	0.19	10%
500	Acetone	500	10%	0.22	10%
511	Acetone	510	10%	0.22	10%

Figure 8: Example report from QC Browser

6.2.5. After the new MDLs have been entered into QuantIMS and reviewed, the QA department files the MDL study report in the file cabinet designated for this purpose.

6.3. Calculations.

Equation 1. Standard deviation (SD).

$$SD = \sqrt{\sum_{n=1}^n \frac{(RF_n - \overline{RF})^2}{n-1}}$$

Equation 2. MDL analyte recovery.

$$\% \text{ Recovery} = \left(\frac{\text{Amount found}}{\text{Amount spiked}} \right) \times 100$$

Equation 3. % relative standard deviation (%RSD).

$$\%RSD = \frac{\text{Standard Deviation}}{\overline{RF_i}} \times 100$$

Equation 4. Calculation of MDL.

$$MDL = \text{Standard deviation} \times t_{(n-1, 0.99)}$$

Where:

$t_{(n-1, 0.99)}$ is the one-sided Student's t value appropriate for a 99% confidence level with $n-1$ degrees of freedom. 40CFR part 136, Appendix B provides critical values of t for the number of replicates supported by this application.

Equation 5. Calculation of spike-to-MDL ratio. Note that both values must have the same units.

$$\text{Spike - to - MDL ratio} = \frac{\text{Spike amount}}{MDL}$$

7. REFERENCES

7.1. References

- 7.1.1. QA Policy CA-Q-S-006, Method Detection Limit Studies, current version.
- 7.1.2. SOP NC-QA-016, Supplemental Practices for DoD Project Work, current version.
- 7.1.3. 40CFR part 136, Appendix B
- 7.1.4. TestAmerica North Canton Quality Assurance Manual (QAM), current version
- 7.1.5. Revision History

<u>Historical File:</u>	Revision 3: 10/04/00
	Revision 4: 11/28/04
	Revision 5: 04/25/07

APPENDIX

2/12/07

Corporate Appendix B

DoD Program Requirements for Limits of Detection (LOD)

- 1.0 The information given in this Appendix is based on the Department of Defense Quality Systems Manual for Environmental Laboratories (QSM), Final Version 3, and the Air Force Quality Assurance Project Plan (AFCEE QAPP), Final Version 4.0. For the purposes of this discussion, the terms LOD and MDL are used interchangeably.
- 2.0 As stated in Grey Box D-18 of the QSM, "Requirements established in 40 CFR 136B are the baseline source of information for determining MDLs." However, the DoD programs impose other requirements that must also be considered:

		Acceptance Limit
Method Blank	Routine compounds	$\leq \frac{1}{2}$ RL
Method Blank	Common lab contaminants	$\leq$ RL
Initial Calibration Blank (ICB)	Metals	$\leq 2 * \text{MDL}$
Continuing Calibration Blank (CCB)	Metals	$\leq 2 * \text{MDL}$
Reporting Limit (RL)	QSM	$\geq 3 * \text{MDL}$
Reporting Limit (RL)	AFCEE QAPP	$\geq 2 * \text{MDL}$

At a minimum, at least for metals methods, the detection limit should be set to a concentration with a high degree of confidence (e.g., 95%) that the detection limit is greater than the 95% confidence limit from long-term blank results. If the 95% confidence limit for long-term blanks is greater than the value obtained from the 40CFR136B study, then the

DoD detection limit should be elevated to the concentration based on blanks. An example is shown in Section 5.0 below.

3.0 Other requirements for DoD MDL studies include the following:

- 3.1 MDL Frequency – There are two options: 1) MDL studies can be run annually or 2) only an initial MDL study is required if the lab chooses to perform quarterly MDL verifications.
- 3.2 Media - A detection limit study must be performed for each analyte and matrix. For water analysis, reagent water is used. For analysis of organic compounds in solids, Ottawa Sand or sodium sulfate is used. For inorganic analytes in solids, Teflon chips or glass beads are used to simulate the soil matrix.
- 3.3 Detection limit studies shall include all preparatory and cleanup methods routinely used on samples.
- 3.4 Instruments - It is acceptable to perform full MDL studies on each instrument, and then use the least-sensitive MDL per compound. It is also acceptable to run the replicate MDL standards on all instruments, as suggested in Appendix A, as long as the calculated MDLs are successfully verified on each instrument as discussed in Appendix B, 4.0 below. Note that for initial DoD MDL/MDLV studies the first approach is more likely to avoid the problem of having to adjust MDLs upward due to subsequent quarterly MDL verifications (MDLVs).
- 3.5 QC Samples Included with MDL Studies – If MDLs are processed independently of client samples, a method blank must be processed together with the spiked aliquots for the MDL determination. A laboratory control sample is not required when only processing spiked aliquots for the MDL determination.
- 3.6 MDL Evaluation - The ratio between the mean recovered concentration and the calculated MDL should be between 1 to 5 for reagent water matrix (some marginal outliers allowed) and should be between 1 to 10 for other matrices. If the ratio for any target analyte is outside the acceptable range, the spiked concentration should be adjusted and the MDL studies repeated. If the laboratory is having difficulty meeting these ratio tests, it may be an indication that the original MDL study was not sufficiently representative of long-term performance for all instruments.

4.0 MDL Verifications (MDLVs)

- 4.1 MDLVs are performed concurrently with or immediately after each MDL studies. In addition, as described in 3.1 above, the lab can opt to run quarterly MDLVs in lieu of annual MDL studies.
- 4.2 MDLVs are prepared at approximately 1-2 times the MDL concentration in the same media as was used for the original MDL study, and are subject to the entire preparation and analysis process. For methods with a large number of analytes and a wide range of MDL concentrations, this will require processing MDLVs at multiple concentrations. For example, MDLVs for Method 8270 must often be prepared at as many as five concentrations.

- 4.3 MDLVs are performed on every instrument that will be used for DoD work.
- 4.4 Peaks for the MDLV standards must be integrated in the same manner as are samples. It is not acceptable to finesse the MDL to lower levels using special manual integrations.
- 4.5 If an MDLV standard is not detected at the first concentration, repeat the verification at approximately twice the previous concentration. The doubling process is repeated as necessary. The final verified MDL is the last concentration detected.

5.0 Detection Limits Adjusted for Long-Term Blanks

- 5.1 For any tests that frequently produce positive blank results, it is advisable to determine if detection limits need to be adjusted for long-term blanks. For metals analyses, it is required in order to meet the DoD ICB and CCB requirements. In these situations, the final verified MDL used for reporting purposes should be greater than long-term blank concentration. If not, the MDL is elevated to the 95% upper confidence limit of long-term blank results.

$$95\%UCL = m + t * s$$

where:

m = mean concentration for uncensored blank results

s = standard deviation

$t_{(n-1, \text{one-tail}, 1-\alpha=0.95)}$ is the students t value appropriate for a 95% upper confidence level and n-1 degrees of freedom:

Student's t, Single-tail, 95% Upper Confidence Limit Table

Number of Replicates	Degrees of freedom (n-1)	Students t value
21	20	1.725
31	30	1.697
41	40	1.684
51	50	1.676
61	60	1.671
71	70	1.667
81	80	1.664

91	90	1.662
101	100	1.660
121	120	1.658
8	8	1.645

5.2 DoD Detection Limit Example

A single-day aluminum MDL study was performed on each of four ICP instruments:

Instrument # 16 → 10.9 ug/L

Instrument # 20 → 6.5

Instrument # 21 → 4.4

Instrument # 22 → 0.71

The highest aluminum MDL for all instruments = 10.9 ug/L

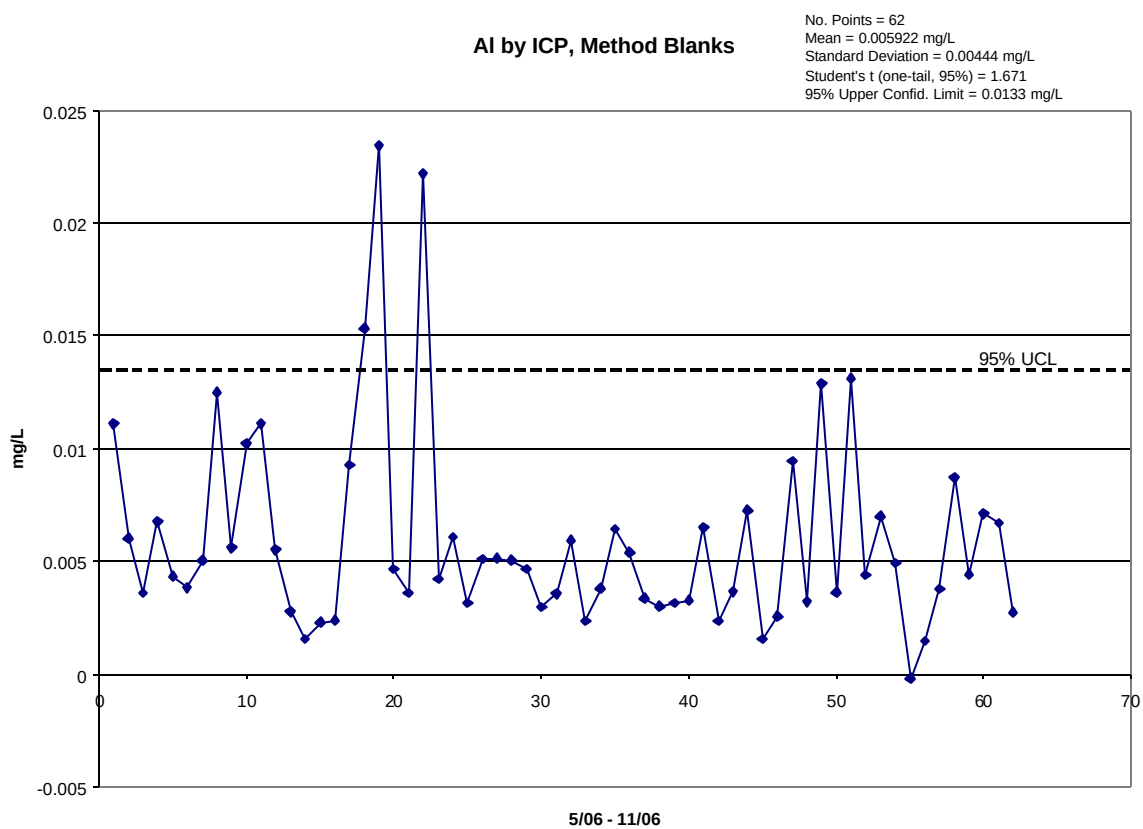
The 95% upper confidence limit was calculated using six months of aluminum data (see chart that follows):

95% UCL = 13 ug/L

The long-term blank is higher than the worst-case MDL. Therefore,

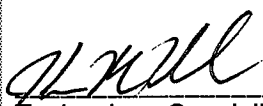
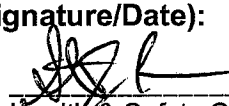
DoD MDL = 13 ug/L

LONG-TERM BLANK DATA EXAMPLE



Title: SAMPLE RECEIVING

[Method: None]

Approvals (Signature/Date):			
	<u>6/30/08</u>		<u>6/30/08</u>
Technology Specialist	Date	Health & Safety Coordinator	Date
	<u>6/30/08</u>		<u>6/30/08</u>
Quality Assurance Manager	Date	Laboratory Director	Date

This SOP was previously identified as SOP No. NC-SC-0005, Rev 6.4, dated 10/02/06

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TABLE OF CONTENTS

1	<i>Scope and Application</i>	3
2	<i>Summary of Method</i>	3
3	<i>Definitions</i>	3
4	<i>Interferences</i>	3
5	<i>Safety</i>	3
6	<i>Equipment and Supplies</i>	5
7	<i>Reagents and Standards</i>	5
8	<i>Sample Collection, Preservation, and Storage</i>	5
9	<i>Quality Control</i>	5
10	<i>Calibration and Standardization</i>	5
11	<i>Procedure</i>	5
12	<i>Data Analysis and Calculations</i>	13
13	<i>Method Performance</i>	13
14	<i>Pollution Prevention</i>	13
15	<i>Waste Management</i>	13
16	<i>References</i>	14
17	<i>Miscellaneous</i>	14

1. SCOPE AND APPLICATION

- 1.1. It is the responsibility of the Sample Receiving Department personnel to perform the procedures described herein in full compliance with this SOP.
- 1.2. It is the responsibility of the Laboratory Director, QA Manager, Operations Manager, and Departmental Supervisor of the facility to assure that the procedures described are performed in full compliance with this SOP. It is also their responsibility to supply adequate training, materials, and equipment to enable personnel to perform this SOP correctly.
- 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.

2. SUMMARY OF METHOD

- 2.1. Not applicable

3. DEFINITIONS

- 3.1. Refer to the glossary in the TestAmerica North Canton Quality Assurance Manual (QAM), current version.

4. INTERFERENCES

- 4.1. Not applicable.

5. SAFETY

- 5.1. Procedures shall be carried out in a manner that protects the health and safety of all TestAmerica associates.
- 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 are worn for tasks that present a strong possibility of getting cut. If personnel are required to perform any portion of the procedure in laboratory areas, appropriate personal protective equipment (PPE) and precautions must be utilized. Disposable gloves that have been contaminated will be removed and discarded.
- 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 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/Unusual Hazards
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.
Sodium Hydroxide	Corrosive	2 mg/m ³ - Ceiling	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	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 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

- 5.4. Exposure to chemicals must be maintained as low as reasonably achievable. If samples are known to be hazardous, they are opened, transferred, and prepared in a fume hood, or under other means of mechanical ventilation, where possible. Contact the EH&S Coordinator if this is not possible. Solvent and waste containers will 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 associate. The situation must be reported immediately to the EH&S Coordinator and the Laboratory Supervisor.

6. EQUIPMENT AND SUPPLIES

- 6.1. Thermometers
- 6.2. PPE such as gloves, lab coats, safety glasses, etc.
- 6.3. Utility knives
- 6.4. pH paper
- 6.5. Copier, printer, computer and label generator
- 6.6. Carts

7. REAGENTS AND STANDARDS

- 7.1. Not applicable

8. SAMPLE COLLECTION, PRESERVATION AND STORAGE

- 8.1. Not applicable

9. QUALITY CONTROL

- 9.1. Nonconformance and Corrective Action
 - 9.1.1. 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. Not applicable

11. PROCEDURE

- 11.1. Receiving and Unpacking Samples
 - 11.1.1. Any deviations from this procedure must be documented as a nonconformance, with a cause and corrective action described.
 - 11.1.2. The procedures listed in this document describe the responsibilities of Sample Receiving personnel in ensuring that data is transmitted correctly from the client to all personnel involved with sample analysis and review.

- 11.1.3. Samples typically arrive packed in coolers or foam boxes. Upon receipt, the coolers are triaged based on rush status (TAT: turn-around time) and expirable tests listed on the Chain-of-Custody (COC). Refer to Appendix I for a listing of expirable tests.
- 11.1.4. The following flagging system is used to show the priority level associated with triaged samples:
- 24 hour TAT – flag with red folder
 - 48 hour TAT – flag with blue folder
 - 72 hour TAT – flag with yellow folder
 - 1 week TAT – flag with green folder
 - Encore or low-level mercury samples – flag with orange paper with “Encore” or “LL Hg” written on it
- 11.1.5. If the COC lists expirable tests, the cooler is marked with a manila folder.
- 11.1.6. Rush and expirable coolers are to be unpacked first and logged as soon as possible. The lab groups are to be notified with any rush TAT requiring 72 hours or less. Samples requiring a standard TAT and/or no expirable tests are given lowest priority.
- 11.1.7. The following information is documented on the Cooler Receipt Form (CRF). Refer to Appendix II for a current version.
- 11.1.7.1. Client name
- 11.1.7.2. Project name or number (if known)
- 11.1.7.3. Signature of Sample Receiving Technician
- 11.1.7.4. Date cooler received and opened
- 11.1.7.5. Method samples were received (overnight courier, client drop-off, or other means)
- 11.1.7.6. Cooler ID number (if known)
- 11.1.7.7. Type of container received (TestAmerica cooler, client cooler, foam box)
- 11.1.7.8. Presence of the custody seals on the outside of the cooler
- 11.1.7.9. Presence of custody seals on bottles
- 11.1.7.10. Presence of the custody papers (i.e., COC)

- 11.1.7.11. Verification that custody papers were properly filled out (ink, signed, match labels)
- 11.1.7.12. Verification that custody papers were relinquished by the client
- 11.1.7.13. Presence of a packing slip
- 11.1.7.14. Presence and type of packing material information
- 11.1.7.15. Cooler temperature upon receipt. The temperature of the cooler is taken by an IR gun. The IR gun is directed at the label (minimizes laser reflection) of a sample bottle or temperature vial, whichever best reflects the cooler temperature. According to federal regulations, the temperature upon receipt should be $\leq 6^{\circ}\text{C}$. The Project Manager (PM) is contacted (and the anomaly is narrated) when the temperature is $> 6^{\circ}\text{C}$ or if samples are frozen.
- 11.1.7.16. Condition of bottles upon receipt (good condition, broken, etc.)
- 11.1.7.17. Complete bottle labels (date, time, client ID)
- 11.1.7.18. Information on bottle labels and tags agree with custody papers
- 11.1.7.19. Verification that the correct bottles were received for the tests indicated (refer to Appendix III)
- 11.1.7.20. Volatile (VOA) vials were checked for the presence of air bubbles. If vials have bubbles exceeding 6 mm in diameter, it is narrated and the Project Manager (PM) is contacted.
- 11.1.7.21. Verification that sufficient amount of sample was received in order to perform the tests listed on the COC.
- 11.1.7.22. Verification of pH for preserved samples (except Volatiles and TOC) upon receipt. This is done by removing sample lids and using a droplet of sample from in the lid to test the pH. The pH's are then recorded on the CRF. The pH paper strips are then discarded.
 - 11.1.7.22.1. Purchased prepared vials of preservatives (refer to Appendix IV) are used if samples are not at the correct pH. The pH is adjusted by adding the appropriate preservative in 5 mL increments up to a maximum of 20 mL per liter of sample or unless there is a reaction. The pH adjustment and final pH are noted on the CRF. The Lot Number of the pre-made preservative is noted on the CRF. It is the responsibility of the Sample Receiving group to change the lot number when a new shipment arrives and to document when a new box is opened.

11.1.7.22.2. The concentrations of the preservatives used are as follows:

4N Sodium Hydroxide

1N Zn Acetate

1:1 HCL (18%)

1:4 HNO₃ (18%)

1:2 H₂SO₄ (33%)

11.1.7.23. The presence or absence of a Trip Blank and if VOA samples were received.

11.1.7.24. Documentation (name of PM, date, name of Sample Receiving technician, method used to contact PM) that the PM was notified of any anomalies associated with receipt of a project.

11.1.8. The Sample Receiving technician removes all sample containers. Any broken, leaking, or dirty sample containers are to be placed inside the fume hood. Dirty sample containers are to be cleaned appropriately, so as not to contaminate the sample storage area.

11.1.9. Coolers emitting strong vapors/fumes when opened will be taken to the High Hazard Room and unpacked in either of its hoods.

11.1.9.1. Any problems concerning exposure while unpacking samples must be immediately reported to the Group Leader and/or the EH&S Coordinator.

11.1.10. Volatile sample(s) suspected (e.g., odor) or known (client information or site history) to be high in volatile concentration is stored in a separate designated volatile area.

11.1.11. The Sample Receiving technician examines all documents and compares information on the sample container labels to the COC. All discrepancies are documented on the CRF.

11.1.12. Problems or discrepancies that compromise sample integrity (such as limited sample volume, sample identification which cannot be determined from the COC, incorrect pH levels, or preservatives, if known, or broken leaking samples) are reported to the PM. The PM will advise Sample Receiving on how to proceed.

11.1.13. Expirable tests (hold time 48 hours or less) must be written on the top of the bottles from which they are to be analyzed. If more than one method exists for the analysis, the method must also be written.

11.1.13.1. Sample containers are placed in the red bin designated for expirables in the Sample Receiving room. The work order or sample ID must be recorded on the expirable logsheet along with the record of the test to be run, special method, if necessary, and the initials of the person relinquishing the sample. The Wet Chem Lab group checks this bin throughout the day, and is responsible for signing out the sample container when they take it.

11.1.14. Samples requiring a Total Solid (TS) result are split off for analysis. Splitting these samples is the responsibility of the Sample Receiving group. A small representative portion (approx.

5-10 grams) from each sample is placed into a small plastic snap-top container designated for the TS analysis. If the TS container is not labeled with a LIMS label when the aliquot is removed, the TS container must be labeled with handwritten client ID. Containers designated for volatiles analyses (VOC) must not be opened in Sample Receiving. If only one container is received and volatiles are requested, the Receiving group must **not** split off because of possible contamination or possible lost volatiles. An empty TS container with a LIMS label is given to the VOC Analysis group for each solid. The VOC group will aliquot for the TS when they open the container for analysis. The TS plastic containers are placed in baggies according to lot number, and are stored in a box inside the walk-in cooler door.

11.1.15. When samples need to be composited, the following procedure is followed, unless there are specific instructions from the client.

11.1.15.1.1. Equal aliquots are weighed from each container and mixed thoroughly and transferred to a new container.

11.1.15.1.2. The amount aliquoted (in grams) is recorded on the Cooler Receipt Form.

11.1.16. The Sample Receiving technician who unpacked the cooler must sign the COC in the appropriate place.

11.1.17. Samples received after hours are signed for by a TestAmerica North Canton employee and placed in the walk-in cooler to be processed the following business morning.

11.2. Sample Log-In

11.2.1. Samples are logged in LIMS after unpacking has been completed, and all anomalies have been documented.

11.2.2. All COCs are photocopied, and given to the associated PM, who in turn, provides the appropriate quote to be used for log-in.

11.2.3. A quote must reflect what is on the Chain-of-Custody. Any discrepancies must be resolved by the PM. Projects are generally not logged until the PM provides a quote. However, if it is a routine project and the quote is known, log-in can proceed.

11.2.4. In the event a project cannot be logged due to discrepancies, all associated paperwork is placed into a black folder. All samples and the black folder are put into cold storage. These projects are recorded on the dry-erase board posted on the walk-in cooler door.

11.2.5. Log-in is initiated by retrieving the paperwork (including a photocopy of the COC or an email from the PM, which lists the TAT and quote number) from the unpacked projects on the counters. The following information is included at log-in: analytical due date, report due date, type of package (expanded deliverable or standard), client sample ID, sample collection date and time, sample matrix (example: WW = wastewater, WG = groundwater, SO = solid), sample type (example: Normal = N1, Trip Blank = TB, Rinse Blank = RB1), and the analyses requested on the COC.

11.2.5.1. 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 No. A8C010121 is described as follows:

A - TestAmerica location where the samples were received
(A = North Canton, C = Pittsburgh, etc.)

8 - Last digit of the year (i.e., 2008)

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.

<u>Client ID</u>	<u>Lot No. – Sample No.</u>	<u>Analysis</u>
AB100	A8E100101-001	PCB
AB101	A8E100101-002	PCB
AB102	A8E100101-003	PCB
AB103	A8E100101-004	PCB

11.2.6. 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.

11.2.7. Work Order Numbers: As each parameter is logged, a random unique 8-digit Work Order number is generated by LIMS. For example, Work Order No. 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.

11.2.8. 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, CREE4-005. These numbers are used to track sample containers through the analytical process.

11.3. Labeling

11.3.1. After log-in is completed, the sample containers are labeled. All labels contain the following information: lot number, sample number, Work Order number, bottle count number, number and type of bottles, client sample ID, client code, date/time of sample collection, and the type of project (example: RCRA) (refer to Appendix V). Labeling samples is a critical step in the log-in process. Care must be taken to ensure client sample ID's and lab ID's match, sample storage locations are correct, the container count and type is correct. Mislabeling can result in incorrect text being run on inappropriate samples.

11.3.1.1. Labels that read "Caution-Use Hood!" shall be affixed to all containers for a given sample that are thought to be a safety hazard (for example, high in contaminants, flammable, etc.), or known to emit noxious fumes or odors. The Sample Receiving group is notified of potential hazards by the PM, COC, quote, or client.

11.3.1.1.1. Samples that are known or expected to contain high concentration of Cyanide (250 ppm or more) or Sulfide (500 ppm or more) need to be unpacked in a fume hood. The Sample Receiving group must put a special sticker on these sample bottles indicating to the Lab Groups that the samples are high in either Cyanide or Sulfide so the Lab Groups can take the necessary safety precautions.

11.4. Storage

11.4.1. Once all sample containers have been properly labeled, the Sample Receiving technician will place the samples into the proper storage locations. These locations are as follows:

- Organic extractable samples are placed in the walk-in refrigerators located in Sample Receiving.
- Volatile samples are stored in the double-door refrigerator located in the Sample Return area.
- Samples known or suspected to be hazardous are stored in a cage in the walk-in cooler, in a fume hood in the High-Hazard Room, and/or in a fume hood in the Volatile rooms.
- Inorganic samples are stored in the walk-in refrigerators located in Sample Receiving.
- Preserved metal samples are placed in a non-refrigerated room. Metals samples that need to be lab filtered and/or preserved are stored in the walk-in cooler. When advised of a Navy project, metal samples are stored in the walk-in cooler.

11.5. Paperwork

- 11.5.1. All paperwork (COC, CRF, LIMS log-in sheets) associated with a project is clipped together and referred to as a Summary.
- 11.5.2. For clients who request a show of sample transfer from sample receipt to storage, a sample control record is printed (see Appendix VI). TestAmerica LIMS will generate this record for Expanded Deliverable designated samples. This record is referred to as an Internal Chain-of-Custody (COC).
- 11.5.3. The Internal COC is attached to the Summary.

11.6. Bottle Returns

- 11.6.1. After analysts have retrieved and analyzed samples, the containers are returned to Sample Receiving for storage and/or disposal documentation.
- 11.6.2. The Sample Return area is located in a hallway behind the walk-in-cooler.
- 11.6.3. A "Sample Custodian Removal Request Form" accompanies the samples (see Appendix VII).
- 11.6.4. The Sample Receiving technician records the return on the Removal Request Form and re-enters custody of the bottles to the Sample Receiving area. The Sample Receiving technician initials and dates the Return form.
- 11.6.5. When sample bottles are consumed in the analysis process, the empty container is returned. The letter "C" is marked on the Removal Request Form beside the appropriate sample to indicate a consumed sample.
- 11.6.6. Samples returned after Sample Receiving Dept. has left for the day are placed in the walk-in cooler by the analyst. Sample Receiving records as received on the next working day.
- 11.6.7. All Removal Request Forms remain in the Sample Return area for approximately one month. After this time, the forms are boxed and stored for seven years.

11.7. Subcontracting of Samples

- 11.7.1. Samples that are logged for parameters performed at other laboratories are subcontracted to these facilities (including other TestAmerica labs).
- 11.7.2. A Sample Analysis Requisition (SAR) is printed for these samples upon completion of the log-in process (see Appendix VIII).
- 11.7.3. The SAR contains information necessary for sample analysis. The original form accompanies the samples to the subcontracted laboratory, and a copy is attached to the Summary. The SAR form must have a relinquished signature with a date and time. Any additional information necessary for sample analysis must be handwritten on the form (e.g. list of

compounds, homogenizing of samples, limited quantity, etc.). In order to track subcontracted samples, the lab Purchase Order number on the SAR form must be recorded in the subcontracted sample PO book located in the Sample Receiving log-in area. (Appendix IX).

12. DATA ANALYSIS AND CALCULATIONS

12.1. Not applicable

13. METHOD PERFORMANCE

13.1. Training Qualifications

13.1.1 One time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using a Nonconformance Memo and approved by a Technical Specialist and QA Manager. The Nonconformance Memo shall be filed in the project file.

13.1.2 Any unauthorized deviations from this procedure must also be documented as a nonconformance, with a cause and corrective action described.

13.1.3 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.

13.1.4 The only personnel authorized to execute this SOP are those in the Sample Receiving Dept.

14. POLLUTION PREVENTION

14.1. This method does not contain any specific modifications that serve to minimize or prevent pollution.

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 Safety Manual for "Waste Management and Pollution Prevention".

15.2. PPE required for purging sample wastes include a lab coat, face shield, and gloves.

15.3. All samples may be disposed of 30 days after the report date except for those samples associated with special client retention. Shelves are purged in chronological order. All lots on a specific shelf must be noted. If a lot can be disposed, a disposal date must be recorded.

15.4. Disposal dates are recorded on this print-out. When clearing shelves, samples that cannot be disposed of because of special client retention are boxed or stored on carts. Stored samples must have a specific

date listed in which samples can be disposed of, or a note that indicates "SAVE" and client name or reason.

- 15.5. A print-out of all lots assigned to certain shelves in each storage location can be obtained from LIMS.
- 15.6. Solid samples that are non-regulated waste are placed in a cubic yard container for disposal.
- 15.7. Regulated solid waste is placed in the "MIXED WASTE" container.
- 15.8. Water samples designated for disposal are placed on carts and disposed of in Sample Receiving. Acidified samples are poured into a drum and neutralized under a fume hood in Sample Receiving.
- 15.9. Solvent waste must be disposed of in clearly labeled waste cans.

16. REFERENCES

16.1 References

16.1.1 TestAmerica North Canton Quality Assurance Manual (QAM), current version

16.1.2 TestAmerica Corporate Safety Manual, M-E-0001, and TestAmerica North Canton Facility Addendum and Contingency Plan, current version

16.2 Associated SOPs and Policies, current version

16.2.1 QA Policy, QA-003

16.2.2 Supplemental Practices for DoD Project Work, NC-QA-0016

16.3 Revision History

<u>Historical File:</u>	Revision 6: 09/02/99	Revision 6.3: 04/20/04
	Revision 6.1: 06/14/01	Revision 6.4: 10/02/06
	Revision 6.2: 10/06/03	

17. MISCELLANEOUS

17.1 Appendices

17.1.1 Appendix I – Example of Expirables

17.1.2 Appendix II – Example of Cooler Receipt Form

17.1.3 Appendix III – Example of Sample Containers, Preservatives, Holding Times

17.1.4 Appendix IV – Example of Preservatives

17.1.5 Appendix V – Example of Label

17.1.6 Appendix VI – Example of Internal Chain-of-Custody

17.1.7 Appendix VII – Example of Sample Custodian Removal Request

17.1.8 Appendix VIII – Example of Sample Analysis Requisition

17.1.9 Appendix IX – Example of Sub-Work Tracking

Appendix I – Example of Expirables

TESTAMERICA NORTH CANTON EXPIRABLES

24-HOUR HOLD TIME:

pH – WATER (9040/4500/150.1—must be on bottle)

*Cr⁺⁶ – WATER (7196/3500—must be on bottle)

FERROUS IRON

RESIDUAL CHLORINE

DISSOLVED OXYGEN (DO)

CORROSIVITY – WATER

48-HOUR HOLD TIME:

BOD/CBOD

*NO₂ – IC (300/9056) or TRACCS (353)

*NO₃ – IC (300/9056) or TRACCS (353)

*OPO₄ – IC (300/9056) or MANUAL (365/4500)

ALKALINITY – TOTAL, CARB, BI-CARB, HYDROXIDE

TURBIDITY

CORROSIVITY – SOLID

SETTLEABLE SOLID

*CR⁺⁶, NO₂, NO₃, AND OPO₄ ARE VERY IMPORTANT.
THESE SHOULD BE DONE FIRST!!!

Appendix III – Example of Sample Containers, Preservatives, Holding Times

PRESERVATIVES, CONTAINERS, AND VOLUMES

Parameter	Container	Preservative _{1,2}	Volume	Parameter	Container	Preservative _{1,2}	Volume
601/602	G	HCl	3x40 mL	PCB	G	None	2 L
Acidity	P	None	250 mL	Pesticides	G	None	2 L
Alkalinity (Sep)	P	None	250 mL	Pesticides + PCBs	G	None	2 L
Amenable Cyanide	P	NaOH	250 mL	pH	P	None	50 mL
Ammonia Nitrogen (NH ₃)	P	H ₂ SO ₄	500 mL	Phenols	G	H ₂ SO ₄	1 L
Asbestos	P	None	250 mL	PNA/PAH	G	None	2 L
Bicarbonate	P	None	250 mL	R. Chlorine	P	None	100 mL
BNA + Dioxin	G	None	2 L	Radiological Alpha, Beta, Radium	P	HNO ₃	4 L
BNAs	G	None	2 L	Reactive Cyanide	P	None	1 L
BOD	P	None	250 mL	Reactive Sulfide	P	None	1 L
Bromide (Br)	P	None	250 mL	Settleable Solids	P	None	1L
BTEX & MTBE	G	HCl	3x40 mL	Silica	P	None	250 mL
BTEX 8021	G	HCl	3x40 mL	Sulfate	P	None	250 mL
Carbonaceous BOD	P	None	250 mL	Sulfide	P	Zn Acetate & NaOH	1 L
Carbonate	P	None	250 mL	Sulfite	P	None	250 mL
Chemical Oxygen Demand	P	H ₂ SO ₄	250 mL	Surfactants (MBAS)	P	None	250 mL
Chloride (Cl)	P	None	250 mL	T. Coliform	P	None	125 mL
Chromium, ⁶⁺	P	None	250 mL	TDS	P	None	250 mL
COD	P	H ₂ SO ₄	250 mL	TKN	P	H ₂ SO ₄	1L
Color	P	None	50 mL	TON	P	H ₂ SO ₄	1 L
Conductivity	P	None	250 mL	Total Cyanide	P	NaOH ³	250 mL
Corrosivity	P	None	250 mL	Total Organic Carbon (TOC)	G	HCl	2 x40 mL
Dissolved Metals*	P	HNO ₃	1 L	Total Organic Halogens	G	H ₂ SO ₄	250 mL
Dissolved Oxygen	G	None	300 mL	Total Phosphorus	P	H ₂ SO ₄	250 mL
Elemental PO ₄	G	None	250 mL	Total Solids	P	None	250 mL
Fecal Coliform	P	None	125 mL	TPH - Diesel (Ext.)	G	None	2 L
Flashpoint	G	None	100 mL	TPH - Gasoline (P&T)	G	HCl	2x40 mL
Fluoride	P	None	250 mL	TPH-GC	G	None	2 L
Formaldehyde	G	None	500 mL	TRPH - IR 418.1	G	HCl	2 L
Free Cyanide	P	NaOH	250 mL	TSS	P	None	250 mL
Glycols 8015	G	None	2x40 mL	Turbidity	P	None	250 mL

PRESERVATIVES, CONTAINERS, AND VOLUMES

Parameter	Container	Preservative _{1,2}	Volume	Parameter	Container	Preservative _{1,2}	Volume
Hardness	P	HNO ₃	250 mL	TVS	P	None	250 mL
Herbicides	G	None	2 L	VOC	G	HCl	3x40 mL
Metals	P	HNO ₃	1 L	VOC 601	G	HCl	3x40 mL
Nitrate	P	None	250 mL	VOC 602	G	HCl	3x40 mL
Nitrate/Nitrite	P	H ₂ SO ₄	250 mL	VOC 624	G	HCl	8x40 mL
Nitrite	P	None	250 mL	VOC 624	G	HCl	3x40 mL
Oil & Grease	G	H ₂ SO ₄	1 L	VOC 8260	G	HCl	3x40 mL
OPPs	G	None	2 L	VOC and VOA	G	HCl	3x40 mL
Orthophosphate	P	None	250 mL				

Notes:

* Filtered in field

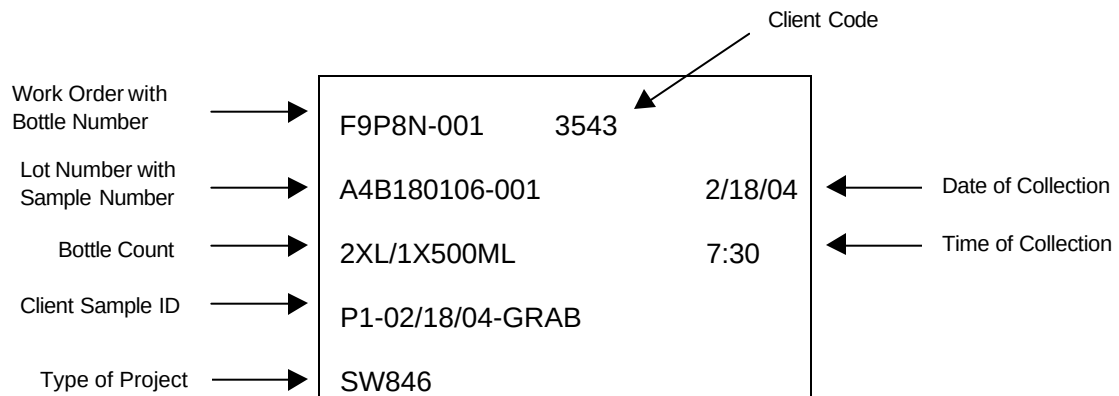
¹ HCl, HNO₃, and H₂SO₄ to pH < 2. NaOH to pH > 12² Temperature = ≤ 6°C except for aqueous metals

Appendix IV – Example of Preservatives

PRESERVATIVES

NITRIC ACID	< 2	HNO ₃	Add Nitric Acid to Contents After Filling
SULFURIC	< 2	H ₂ SO ₄	Add Sulfuric Acid to Contents After Filling
SODIUM HYDROXIDE	> 12	NAOH	Add Sodium Hydroxide to Contents After Filling
HYDROCHLORIC ACID	< 2	HCl	Add Hydrochloric Acid to Bottle Prior to Filling
			Add Zinc Acetate to Contents After Filling
ZINC ACETATE/SODIUM	> 9	ZNACE/NAOH	Add Sodium Hydroxide to Contents After Filling

WI-NC-004A-022007

APPENDIX V – Example of Laboratory-Generated Bottle Label

Appendix VI – Example of Internal Chain-of-Custody**TestAmerica Laboratories, Inc.****Sample Control Record****Client:****Lot #:****Case Number/SDG:****Storage Location:**

Laboratory Sample ID	Transferred By	Date	Entered	Removed	Reason	Date Returned

Appendix VII – Example of Sample Custodian Removal Request

PSR024 5/23/08 6:47:31 MT SAMPLE CUSTODIAN REMOVAL REQUEST PAGE 001

REQUESTED BY:

METHOD: B2 Fluoride (4500F-C, ISE, SM18)

STORAGE LOCATION	WORK ORDER #	PICKED CNTR#	CONTROL #	CLIENT #	ANALYSIS	LOTID	SMP#	SPX	MATRIX DESCRIPTION	QTY RCVD	QTY REQD
C36 S31 MS	KM6HD-1-AQ	007	348422	001610	I-88-B2 ASE140184	001			WATER	8	1
C36 S31 MS	KM6HX-1-AQ		348423	001610	I-88-B2 ASE140184	002			WATER	8	1
C36 S31 MS	KM6HL-1-CL		348424	001610	I-88-B2 ASE140184	003			WATER	24	1
C36 S31 MS	KM6HN-1-AQ		348425	001610	I-88-B2 ASE140184	004			WATER	8	1
C36 S31 MS	KM6HP-1-AQ		348426	001610	I-88-B2 ASE140184	005			WATER	8	1
C36 S31 MS	KM6HR-1-AQ		348427	001610	I-88-B2 ASE140184	006			WATER	8	1
C36 S31 MS	KM6HV-1-AQ		348428	001610	I-88-B2 ASE140184	008			WATER	8	1
C36 S31 MS	KM6HX-1-AQ		348429	001610	I-88-B2 ASE140184	009			WATER	8	1
C54 S35 MS	KM6DL-1-AQ		348430	001610	I-88-B2 ASE150313	002			WATER	8	1
C54 S35 MS	KM6DP-1-CL		348431	001610	I-88-B2 ASE150313	003			WATER	24	1
C54 S35 MS	KM6DW-1-AQ		348432	001610	I-88-B2 ASE150313	004			WATER	8	1
C54 S35 MS	KM6DX-1-AQ		348433	001610	I-88-B2 ASE150313	005			WATER	8	1
C54 S35 MS	KM6D2-1-AQ		348434	001610	I-88-B2 ASE150313	006			WATER	8	1
C54 S35 MS	KM6D3-1-AQ		348435	001610	I-88-B2 ASE150313	007			WATER	8	1
C54 S35 MS	KM6DC-1-AQ		348436	001610	I-88-B2 ASE150313	009			WATER	8	1
C54 S35 MS	KM6DF-1-AQ		348437	001610	I-88-B2 ASE150313	010			WATER	8	1

RELINQUISHED BY

RECEIVED BY

DATE/TIME

***** END OF REPORT *****

Appendix VIII – Example of Sample Analysis Requisition

TestAmerica Laboratories, Inc.
SAMPLE ANALYSIS REQUISITION

Laboratory: (Subcontracting Lab)

Report Package: (Type of Report)

Lab Request: (Sample Rcvg. P.O.)

Need Analytical Report: (Date)

Client Code:

Project Manager:

<u>Sample I.D.</u>	<u>W.O. No.</u>	<u>Client Sample ID</u>	<u>Sampling Date</u>	<u>Analysis Required</u>
Example : A8B010350	F9DED	MW-6	2008-01-31 11:12	WATER, 6010B, Total Lithium - (Subcontr. Lab)

Please use Client Sample ID for report

Call (Project Manager) with questions at 330-497-9396

At TestAmerica North Canton Laboratory

Need detection limit and analysis date included in report.

Shipping Method: **FEDEX**

Please send a signed copy of this form with the report at completion of analysis.

Relinquished by: _____ Date/Time: _____

Relinquished by: _____ Date/Time: _____

Received for lab by: _____ Date/Time: _____

Appendix IX – Sub-Work Tracking

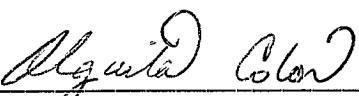
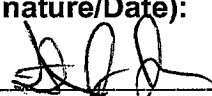
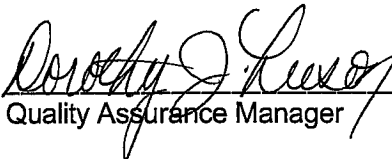
TestAmerica North Canton
Sub-Work Tracking

Tracking No.	Client Code	Lot Number	No. of Samples	Analysis	Lab Sent To	Date Out	Shipment Method	Initials

Title: pH ELECTROMETRIC METHOD

[SW846 Methods 9040B and 9045C, EPA Method 150.1]

Approvals (Signature/Date):

	
Technology Specialist	Health & Safety Coordinator
4/28/08	4/29/08
Date	Date
	
Quality Assurance Manager	Laboratory Director
04/28/08	4/29/08
Date	Date

This SOP was previously identified as SOP NC-WC-0010, Rev 7, dated 03/23/06

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TABLE OF CONTENTS

1. <i>Scope and Application</i>	3
2. <i>Summary of Method</i>	3
3. <i>Definitions</i>	3
4. <i>Interferences</i>	3
5. <i>Safety</i>	3
6. <i>Equipment and Supplies</i>	4
7. <i>Reagents and Standards</i>	5
8. <i>Sample Collection, Preservation, and Storage</i>	5
9. <i>Quality Control</i>	5
10. <i>Calibration and Standardization</i>	6
11. <i>Procedure</i>	7
12. <i>Data Analysis and Calculations</i>	9
13. <i>Method Performance</i>	9
14. <i>Pollution Prevention</i>	10
15. <i>Waste Management</i>	10
16. <i>References</i>	10
17. <i>Miscellaneous (Tables, Appendices, Etc.)</i>	11

1. SCOPE AND APPLICATION

- 1.1. This method is applicable to the determination of pH in waters, wastewaters, and solids. It is based on SW846 Methods 9040B and 9045C and EPA Method 150.1. Method 9040B should be used if the aqueous phase constitutes at least 20% of the total volume of the waste. Method 9045C should be used for measuring pH in soils and waste samples. If water is present, it must constitute less than 20% of the total volume of the sample.
- 1.2. The approximate working range is 1 - 14 pH units. Samples with a pH of < 1 are reported as < 1 .
- 1.3. The associated method codes are PU (9040B), OZ (9045C), and AJ (150.1). The preparation codes are 88 and 1C.
- 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. The pH is determined electrometrically by using an electrode. The pH meter is calibrated with a series of known pH buffers.

3. DEFINITIONS

- 3.1. Refer to the glossary in the TestAmerica North Canton Quality Assurance Manual (QAM), latest version.

4. INTERFERENCES

- 4.1. Samples with very low or very high pH may give incorrect readings on the meter. For samples with a true pH of >10 , the measured pH may be incorrectly low. This error can be minimized by using a low sodium error electrode.

5. SAFETY

- 5.1. Employees must abide by the policies and procedures in the Corporate Safety Manual and this document.

- 5.2. There are no materials used in this method that have a serious or significant hazard rating.
NOTE: This list does not include all materials used in the method. 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.
- 5.3. 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 will be removed and discarded; other gloves will be cleaned immediately.
- 5.4. Exposure to chemicals must be maintained **as low as reasonably achievable**; therefore, unless they are known to be non-hazardous, all samples must be opened, transferred and prepared in a fume hood, or under other means of mechanical ventilation. Solvent and waste containers will be kept closed unless transfers are being made.
- 5.5. The preparation of standards and reagents will be conducted in a fume hood with the sash closed as far as the operation will permit.
- 5.6. It is recommended that neat standards be purchased only as a last resort. The preparation of standards from neat materials and reagents should be conducted in a fume hood with the sash closed as far as the operations will permit.
- 5.7. Standards in solution may be diluted in the open laboratory when syringes and the like are utilized.
- 5.8. 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. pH meter with electrode(s) and temperature compensation
- 6.2. Beakers: various
- 6.3. Top loading balance: Capable of accurately weighing ± 0.01 g
- 6.4. Stir plate and stir bars
- 6.5. Shaker or mechanical tumbler

6.6. Autotitrator

6.7. Centrifuges tubes

7. REAGENTS AND STANDARDS

7.1. Standards

7.1.1. Target Calibration Standards

7.1.1.1. pH 2, 4, 7, 10, and 12 buffers--purchased

7.1.1.2. Fresh buffers are poured and used each working day.

8. SAMPLE COLLECTION, PRESERVATION AND STORAGE

8.1. Samples are not chemically preserved.

8.2. Samples are stored in plastic or glass containers at $4^{\circ}\text{C} \pm 2^{\circ}\text{C}$.

8.3. Samples should be analyzed as soon as possible after sampling, but not to exceed one day after sampling.

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 and Sample Duplicate) 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. Sample Duplicate

9.2.1. 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.

- 9.2.2. Sample duplicates are performed at a frequency of 10% per matrix, and must meet laboratory-specific limits for precision.

9.3. Laboratory Control Sample (LCS)

- 9.3.1. One aqueous LCS must be processed with each analytical 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 commercially available (Environmental Resource Associates or equivalent) control standard will be analyzed. Recovery must be within +/-2% of true value.

9.3.3. Corrective Action for LCS

- 9.3.3.1. If the pH is outside the established control limits the system is out of control and corrective action must occur.

- 9.3.3.2. Corrective action consists of identification and correction of the cause for the out of control situation and reanalysis of all effected samples.

10. CALIBRATION AND STANDARDIZATION

10.1. Initial Calibration

- 10.1.1. Refer to the manufacturer's manual for instrumental calibration.

- 10.1.2. The following procedure is applicable for use with the Orion 320 pH meter.

- 10.1.2.1. If in "Standby" mode, press "Mode". Rinse the electrodes with reagent water and place in the pH 7 buffer. Press "Cal". When 7-4 is displayed, press "yes". Allow to stabilize. When "Ready" light comes on, press "yes".

- 10.1.2.2. Rinse electrode with reagent water, and place in pH 4 buffer. When "Ready" light comes on, press "yes". Record electrode slope on analytical logsheet.

- 10.1.2.3. Calibration Check: Rinse the electrodes and place in the pH 10 buffer.

Allow value to stabilize. The pH should be between 9.95 and 10.05 or recalibration is necessary.

10.1.2.4. After calibration, run a pH 7, a pH 2, a pH 12, then an LCS, allowing the electrode to stabilize for each. Record results on analytical logsheet.

10.1.3 The pH meter should be calibrated daily. The calibration is recorded on the analytical logsheet.

10.1.4 If the pH meter has been turned off, it must be calibrated prior to use.

10.2. Continuing Calibration

10.2.1. A pH 7 buffer is analyzed before analysis and every ten samples to ensure the calibration remains linear.

10.2.2. The pH meter must be recalibrated if the buffer deviates by more than ± 0.05 . If this range is exceeded, re-analyze all samples analyzed since the last pH buffer that met criteria.

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 shall be completely documented using a Nonconformance Memo and is approved by a Technical Specialist and QA Manager. The Nonconformance Memo shall 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.

11.3. Sample Preparation

11.3.1. Waters

11.3.1.1. No preparation necessary for waters and wastewaters.

11.3.2. Solids and Soils

11.3.2.1. Place 10 g (± 0.5 g) of sample in a beaker or other suitable container.

11.3.2.2. Add 10 mL of reagent water and mix for five minutes in a shaker or mechanical tumbler.

11.3.2.3. Allow sample to stand for one hour to allow the solids to settle out.

11.4. Sample Analysis

11.4.1. Manual Procedure

11.4.1.1. Waters

11.4.1.1.1. Place the sample in a clean beaker using a sufficient volume to cover the sensing elements of the electrode(s). Allow the pH to stabilize (swirling or stirring may quicken stabilization). Record the pH on the analytical logsheet. Remove the electrodes from the sample. Rinse and gently dab off the electrodes between each measurement. Store the electrodes in pH 7 buffer when not in use.

11.4.1.1.2. For 9040B – Continuously stir the sample while obtaining a stable reading.

11.4.1.2. Solids

11.4.1.2.1. Immerse the pH electrodes in the supernatant layer of the sample - be careful not to stir up solids. Allow pH to stabilize and record it on the analytical logsheet. Remove and rinse the electrodes between each measurement. Store electrodes in the pH 7 buffer.

NOTE: If the sample contains oil or other substances that will coat or damage the electrodes, the pH should be analyzed by pH - Paper Method.

11.4.2. Automated Procedure

11.4.2.1. Load the appropriate schedule on the autotitrator starting with the pH calibration.

11.4.2.2. Pour a homogenized sample into the centrifuge tubes and place the tubes in the appropriate position on the autosampler. Remember to include a pH 7 buffer check after every ten positions.

11.4.2.3. Start the autotitrator.

11.5. Analytical Documentation

11.5.1. Record all analytical information in the analytical logbook/logsheet, including the analytical data from standards 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 an 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

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 that 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. This method does not contain any specific modifications that serve to minimize or prevent pollution.

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 Safety Manual 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 and alkaline sample waste and exhausted buffer solutions poured down the drain if the pH is between 5 and 10. Any sample waste generated that is not in this pH range is collected in a designated container identified as "Acid Waste".

15.2.1.2. Exhausted soil or oil samples analyzed by the method. The liquid layer is decanted and disposed of in a designated container identified as "Acid Waste". The remaining solid layer is disposed of by placing it in a 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 of TestAmerica North Canton. 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, 3rd Edition, pH Electrometric Measurement, Method 9040B

16.1.2. EPA 600, Methods for Chemical Analysis of Water and Wastes, pH (Electrometric),

Method 150.1

16.1.3. SW846, Test Methods for Evaluating Solid Waste, 3rd Edition, Soil pH, Method 9045C.

16.1.4. TestAmerica North Canton Quality Assurance Manual (QAM), current version

16.1.5. TestAmerica Corporate Safety Manual, M-E-0001, 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-0014

16.2.3. Statistical Evaluation of Data and Development of Control Charts, NC-QA-0018

16.2.4. Supplemental Practices for DoD Project Work, NC-QA-0016

16.3. Revision History

<u>Historical File:</u>	Revision 4.0: 01/04/99
	Revision 4.1: 11/28/00
	Revision 5: 02/05/03
	Revision 6: 10/27/04
	Revision 7: 03/23/06
	Revision 8: 04/30/08

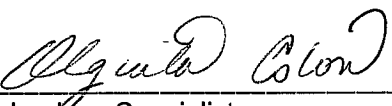
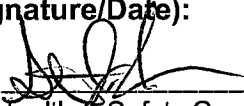
17. MISCELLANEOUS (TABLES, APPENDICES, ETC.)

17.1. Reporting limits

17.1.1. A minimum reporting limit is not listed in LIMS. Units are reported as “No Units”.

Title: CYANIDE AUTOMATED, PYRIDINE-BARBITURIC ACID METHOD

[SW846 Method 9012A, EPA Methods 335.2, 335.4, and Standard
Methods 4500-CN-E, 4500-CN-I, and 4500-CN-G]

Approvals (Signature/Date):			
	5/28/08		5/28/08
Technology Specialist	Date	Health & Safety Coordinator	Date
	5/28/08		5/28/08
Quality Assurance Manager	Date	Laboratory Director	Date

This SOP was previously identified as SOP No. NC-WC-0031, Rev 8, dated 08/24/07

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TABLE OF CONTENTS

1. SCOPE AND APPLICATION	3
2. SUMMARY OF METHOD.....	3
3. DEFINITIONS.....	3
4. INTERFERENCES	3
5. SAFETY.....	3
6. EQUIPMENT AND SUPPLIES	7
7. REAGENTS AND STANDARDS.....	7
8. SAMPLE COLLECTION, PRESENTATION AND STORAGE	9
9. QUALITY CONTROL	9
10. CALIBRATION AND STANDARDIZATION	12
11. PROCEDURE	13
12. DATA ANALYSIS AND CALCULATIONS	15
13. METHOD PERFORMANCE	16
14. POLLUTION PREVENTION.....	17
15. WASTE MANAGEMENT.....	17
16. REFERENCES	17
17. MISCELLANEOUS (TABLES, APPENDICES, ETC...).....	18

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, 335.4 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.
- 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), 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. 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.

5. SAFETY

- 5.1. Employees must abide by the policies and procedures in the Corporate Safety 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 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
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.
Bismuth Nitrate	Oxidizer	None	May cause irritation to the respiratory tract, skin and eyes.
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. .

Sodium Phosphate		None	Inhalation may cause respiratory tract irritation. Can produce delayed pulmonary edema. Causes mild skin and eye irritation. Ingestion 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 increased salivation, choking, vomiting, stomach pains and diarrhea. 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. Should 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.
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.			

- 5.3. 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 Hydrogen 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 will be removed and discarded; other gloves will be cleaned.

- 5.10. Exposure to chemicals must be maintained **as low as reasonably achievable**; therefore, unless they are known to be non-hazardous, all samples must be opened, transferred and prepared in a fume hood, or under other means of mechanical ventilation, where possible. 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 will 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 should 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 $\pm 0.01\text{g}$

7. REAGENTS AND STANDARDS

- 7.1. Reagents
 - 7.1.1. Cadmium carbonate: powder
 - 7.1.2. 1N NaOH: Add 40g of sodium hydroxide pellets (NaOH) to a one liter volumetric flask, and dilute to volume with reagent water.

- 7.1.3. 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.)
- 7.1.4. 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.)
- 7.1.5. 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 $4^\circ\text{C} \pm 2^\circ\text{C}$ in an amber glass bottle. The maximum shelf life is 6 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.

- 7.1.6. 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.

- 7.1.7. Rhodanine indicator, purchased.

- 7.1.8. 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.

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.

<u>Concentration CN- Calibration Standards</u>	<u>mL CN-</u>	<u>Final Volume</u>
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 concentrations or false positive data. The method blank should 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-0032 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 reanalyzed. 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. On-going 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-0032 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 reparation and reanalysis of the batch unless the client agrees that other corrective action is acceptable. For Ohio VAP samples, the LCS must be re-distilled unless the LCS is biased high and the samples are non-detect.
- 9.3.3.3. 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. Matrix Spike/Matrix Spike Duplicate (MS/MSD)

- 9.4.1. 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 (DQO's) may require the use of sample duplicates in place of or in addition to MS/MSD's. 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.
- 9.4.2. A 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) will be distilled and analyzed with every batch. See SOP NC-WC-0032 for distillation instructions.
- 9.4.3. Corrective action for MS/MSDs
 - 9.4.3.1. 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 will include reparation and reanalysis of the batch.

9.4.3.2. If the native analyte concentration in the MS/MSD exceeds 4x 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.

9.4.3.3. If an MS/MSD is not possible due to limited sample volume then a laboratory control sample duplicate (LCSD) should 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.

9.5. QC Acceptance Criteria

9.5.1. Control limits are established by the laboratory as described in NC-QA-018.

9.5.2. Laboratory control limits are internally generated and updated periodically unless method specified. The latest is version easily accessible via LIMs (QC Browser program.).

9.6. Method Detection Limits (MDLs) and MDL Checks

9.6.1. MDLs and MDL Checks are established by the laboratory as described in SOPs CA-Q-S-006 and NC-QA-021.

9.6.2. MDLs are easily accessible via LIMs (QC Browser program).

9.7. Nonconformance and Corrective Action

9.7.1. 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 and 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 $\pm 10\%$, or recalibration is required. The correlation coefficient of the original curve must be ≥ 0.995 , or recalibration is required. The curve must not be forced through the origin.

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 $\pm 15\%$, or recalibration is required. The previous ten samples must be reanalyzed 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 10 samples must be reanalyzed 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 $\pm 10\%$ agreement. Reanalysis will 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, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using a Nonconformance Memo and is approved by a Technical Specialist and QA Manager. The Nonconformance Memo shall 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.

11.3. Sample Preparation

11.3.1. See Cyanide Distillation SOP NC-WC-0032.

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. EPA Method 335.4 says to reflux for one and a half hours.

11.3.2. Sample Preparation Procedure

All 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 should be tested prior to distillation

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 reanalyzed. Dilutions should 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 an 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

$$12.1. \text{ Total Cyanide, mg / L} = \frac{\text{mg / L CN}^- \text{ from printout} \times 50}{\text{mL of sample distilled}} \times D$$

$$12.2. \text{ Total Cyanide, mg / kg} = \frac{\text{mg / L CN}^- \text{ from printout} \times 50}{\text{g of sample distilled.}} \times D$$

$$12.3. \text{ Amenable Cyanide, mg / L} = \text{Total CN}^- \text{ (mg / L)} - \text{Chlorinated CN}^- \text{ (mg / L)}$$

Where:

mg/L = can also be mg/kg

$$D = \text{Dilution Factor} = \frac{\text{Final Volume of Dilution}}{\text{Volume of Sample Distillate Used}}$$

NOTE: Weak Acid Dissociable Cyanide has the same calculations as Total Cyanide

12.4. LCS Recovery:

$$\frac{\text{Instrument Value}}{0.04(\text{true})} \times 100 = \% \text{ Recovery}$$

12.5. CCV Recovery:

$$\frac{\text{Instrument Value}}{0.025(\text{true})} \times 100 = \% \text{ Recovery}$$

NOTE: CCV recovery must be between 85 - 115% for data to be acceptable. If CCV recovery is not within these limits, reanalysis is required 335.4 says 10%.

12.6. MS/MSD Recovery for Waters and solids

$$\frac{A - B}{0.040(\text{true})} \times 100 = \% \text{ Recovery}$$

Where:

A = Instrument value MS/MSD

B = Sample instrument value

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 that 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. This method does not contain any specific modifications that serve to minimize or prevent pollution.

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 Safety Manual 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 lead sulfide. 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. Miscellaneous solid waste contaminated with sample residue, acids, caustics and reagents used in this SOP. A weigh tin is the only type of this waste generated. The tin is rinsed with water and disposed of in a container labeled "Solid Waste".

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 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 Quality Assurance Manual (QAM), current version

16.1.6. TestAmerica Corporate Safety Manual, M-E-0001, 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, SOP, NC-QA-0016

16.2.6. Cyanide Preparation Method, NC-WC-0032

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 reporting limit for solid samples performed only under the Michigan program is 200 µg/kg for solids (2.5 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).

Appendix ICYANIDE 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

$$\text{Cyanide, mg / L} = \frac{(\text{A} - \text{B}) (1000)}{\text{mL Cyanide Solution (10)}}$$

Where:

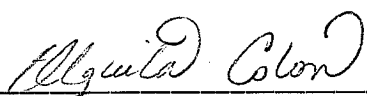
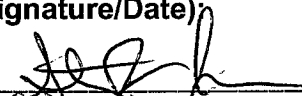
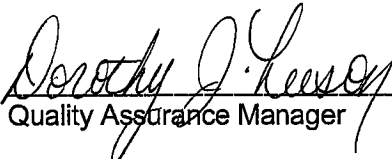
A = mL titrant for standard

B = mL titrant for blank

6. If the cyanide concentration is not 1000 ppm, adjust concentration accordingly.

Title: CYANIDE PREPARATION METHOD

[EPA Methods 335.1, 335.2, 335.4, Standard Method 4500-CN-I,
4500-CN-G, and SW846 Method 9012A]

Approvals (Signature/Date)			
	5/21/08		5/28/08
Technology Specialist	Date	Health & Safety Coordinator	Date
	5/28/08		5/28/08
Quality Assurance Manager	Date	Laboratory Director	Date

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TABLE OF CONTENTS

1. SCOPE AND APPLICATION.....	3
2. SUMMARY OF METHOD.....	3
3. DEFINITIONS.....	3
4. INTERFERENCES	3
5. SAFETY	4
6. EQUIPMENT AND SUPPLIES.....	8
7. REAGENTS AND STANDARDS.....	8
8. SAMPLE PRESERVATION AND STORAGE	11
9. QUALITY CONTROL	11
10. CALIBRATION AND STANDARDIZATION.....	13
11. PROCEDURE.....	14
12. DATA ANALYSIS AND CALCULATIONS.....	17
13. METHOD PERFORMANCE.....	16
14. POLLUTION PREVENTION	18
15. WASTE MANAGEMENT	18
16. REFERENCES.....	18
17. MISCELLANEOUS (TABLES, APPENDICES, ETC...).....	19

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, 335.4, 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), 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 Quality Assurance Manual (QAM), latest 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. Specific selection of reagents may be required to avoid introduction of contaminants.
- 4.2. Oxidizing 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.
- 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.
- 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 Safety 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 Hydrogen 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 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
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.
Bismuth Nitrate	Oxidizer	None	May cause irritation to the respiratory tract, skin and eyes.
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. .

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.
Lead Carbonate	Poison Neurotoxin Irritant Probable carcinogen Reproductive hazard	0.05mg/ m ³ TWA as Lead	Inhalation can cause local irritation of bronchia and lungs and can cause symptoms such as metallic taste in the mouth, chest and abdominal pain. Skin contact can cause local irritation, redness and pain. Can be absorbed through the skin.
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.
Phosphoric Acid	Corrosive	1 mg/m ³ TWA	Inhalation is not an expected hazard unless misted or heated to high temperatures. May cause redness, pain, and severe skin burns. May cause redness, pain, blurred vision, eye burns, and permanent eye damage.
Barbituric Acid	Irritant	Not established	Limited information. Inhalation may irritate respiratory tract. Causes skin and eye irritation. Should 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 will be removed and discarded; other gloves will be cleaned immediately.
- 5.11 Exposure to chemicals must be maintained **as low as reasonably achievable**; therefore, unless they are known to be non-hazardous, all samples must be opened, transferred and prepared in a fume hood, or under other means of mechanical ventilation. 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 will be kept closed unless transfers are being made.
- 5.12 The preparation of standards and reagents will 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
- 6.10. Erlenmeyer flasks: various
- 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
- 6.16. Condensers

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.
- 7.1.5. Sodium Hydroxide, 1.25 N: Add 50 g of NaOH to 900 mL and dilute to 1 liter with reagent water.
- 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: (H₂SO₄), 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: (MgCl₂•6H₂O), reagent grade
- 7.1.10. Magnesium Chloride Solution: Add 510 g MgCl₂•6H₂O 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 (OCl)₂], reagent grade
- 7.1.12. Calcium Hypochlorite Solution: Add 5 g of Ca(OCl)₂ to 100 mL of reagent water.
- 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: (CH₃COOH), glacial reagent grade
- 7.1.16. Zinc Acetate: [Zn(C₂H₃O₂)], 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: [NaC₂H₃O₂•3H₂O] 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

- 7.1.22. Bismuth nitrate [$\text{Bi}(\text{NO}_3)_3$]: Dissolve 30 g of $\text{Bi}(\text{NO}_3)_3$ in 100 mL of reagent water. While stirring, add 250 mL of glacial acetic acid. Stir until dissolved. Dilute to 1 L with reagent water.
- 7.1.23. Cadmium chloride [CdCl_2] – anhydrous : Dissolve 1.0 g CdCl_2 in 90 mL of reagent water. Dilute to 100 mL with reagent water to produce a 10 g/L CdCl_2 solution.

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.

Note: This stock standard may also be purchased.

Note: This stock standard must be standardized prior to use. See SOP NC-WC-0031 Appendix I

- 7.2.2. Secondary Source Cyanide Standard, 1000 mg/L: Follow 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-0031 Appendix I.

7.2.3. Calibration Standards (Water and Solid Matrices)

- 7.2.3.1. If using the 1.25 N NaOH: pipette the appropriate amount of cyanide standard into 100 mL volumetric and add 20 mL 1.25N NaOH to each calibration standard (except the 1.0 mg/L) and bring to volume with reagent water.

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.

<u>Concentration CN-</u>	<u>mL CN-</u>	<u>Final Volume</u>
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 should 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 H₂O and 1.0 g Ottawa sand. The blank must be distilled per Section 11.4.4 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 reanalyzed. 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. On-going 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 11.4.4 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 will be repreparation and reanalysis of the batch unless the client agrees that other corrective action is acceptable

9.3.3.3. 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. Matrix Spike/Matrix Spike Duplicate (MS/MSD)

9.4.1. 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 (DQO's) may require the use of sample duplicates in place of or in addition to MS/MSD's. 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.

9.4.1.1. An MS/MSD consisting of 50 mL or 1.0 g of sample brought up to 50 mL with reagent water and 2.0 mL of the 1.0 mg/L standard (either source) must be distilled with each analytical batch of samples.

9.4.2. Corrective action for MS/MSDs

- 9.4.2.1. 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 reparation and reanalysis of the batch.
- 9.4.2.2. If the native analyte concentration in the MS/MSD exceeds 4x 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.
- 9.4.2.3. If an MS/MSD is not possible due to limited sample volume then a laboratory control sample duplicate (LCSD) should be analyzed. The RPD of the LCS and LCSD must be compared to the matrix spike RPD limits.

9.5 Control Limits

- 9.5.1 Control limits are established by the laboratory as described in SOP, NC-QA-0018.
- 9.5.2 Laboratory control limits are internally generated and updated periodically unless method specified. Control limits are easily accessible via the LIMs (QC Browser program).

9.6 Method Detection Limits (MDLs) and MDL Checks

- 9.6.1 MDLs and MDL Checks are established by the laboratory as described in SOP NC-QA-0021 and CA-Q-S-006.
- 9.6.2 MDLs are easily accessible via the LIMs (QC Browser program).

9.7 Nonconformance and Corrective Action

- 9.7.1 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. The distilled 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 shall be completely documented using a Non-Conformance Memo and is approved by the QA Manager. The Non-Conformance Memo shall be filed in the project file.

11.3 Summary

11.3.1 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.

11.4. Sample Preparation Procedure applicable for use with the Midi distillation unit.

11.4.1. Checking for Interferences

11.4.1.1. 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.**

11.4.1.2. 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.

11.4.1.3. 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.

11.4.2. Complex Cyanide Preparation Procedure [THIS PROCESS IS PERFORMED ONLY WHEN REQUESTED BY THE CLIENT]: Add 100 mL of water sample or 2.0 g of solid sample and 100 mL reagent water to a plastic, sealable container. Add 10 g NaOH pellets. Mix or tumble for 12-16 hours. Allow solids to settle out. Transfer 50 mL of the solution to the distillation tube. Distill sample (See section 11.4.4.1).

Note: If the sample requires an MS/MSD or MS/Dup, double or triple the

volumes as stated above. A blank and LCS should be prepared and analyzed with samples using 10 g NaOH and 100 mL reagent water.

11.4.3. Amenable Cyanide (Chlorinated Aliquot) [THIS PROCESS IS PERFORMED ONLY WHEN REQUESTED BY THE CLIENT]

11.4.3.1. Place 50 mL (waters) or 1.0 g (solids/liquids) into a beaker. 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 $\text{pH} \geq 12$. 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. 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 11.4.4). 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.

Note: If solid samples are being prepared under the Michigan program, the initial solid weight is 2.5 g

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 $\frac{1}{4}$ " 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.

Note: Complex cyanide samples require an additional 2.5 mL of sulfuric acid. Be sure the pH is < 2 .

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 **[THIS PROCESS IS PERFORMED ONLY WHEN REQUESTED BY THE CLIENT.]**
- 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 that it is a Weak Acid Dissociable sample distillate.
- 11.4.6. Cyanide Unit Clean-up
- 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 set-up. Be sure to collect the solids in a screen and dispose of properly.

11.4.6.3. Wash each set-up with hot water. Rinse several times with reagent water.
**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 set-up.

11.4.6.5. Be sure to wash each set up 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/logsheets, 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

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 that 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. Using a Midi Cyanide distillation unit saves time and requires less sample and reagents for use.

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 Safety Manual 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".

- 15.3. 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, and 335.2
- 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 Quality Assurance Manual (QAM, current version
- 16.1.7 TestAmerica Corporate Safety Manual, M-E-0001, and TestAmerica North Canton Facility Addendum and Contingency Plan, current version.

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-0016

16.2.6 Standards and Reagents, NC-QA-017

16.2.7 Cyanide, Automated Method NC-WC-031

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. Method Deviation (9012A, 335.1, 335.2)

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).

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Implementation Date

7-19-07

SOP No. NC-WC-0034

Revision No. 1.1

Revision Date: 06/28/07

Page 1 of 10

TESTAMERICA NORTH CANTON STANDARD OPERATING PROCEDURE

TITLE: FLASHPOINT CLOSED CUP

(SUPERSEDES: Revision 1, Dated 09/25/03)

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TABLE OF CONTENTS

1. SCOPE AND APPLICATION	3
2. SUMMARY OF METHOD	3
3. DEFINITIONS	3
4. INTERFERENCES	3
5. SAFETY	3
6. EQUIPMENT AND SUPPLIES	5
7. REAGENTS AND STANDARDS	5
8. SAMPLE COLLECTION, PRESENTATION AND STORAGE	5
9. QUALITY CONTROL	5
10. CALIBRATION AND STANDARDIZATION	6
11. PROCEDURE	6
12. DATA ANALYSIS AND CALCULATIONS	8
13. METHOD PERFORMANCE	8
14. POLLUTION PREVENTION	8
15. WASTE MANAGEMENT	8
16. REFERENCES	9
17. MISCELLANEOUS (TABLES, APPENDICES, ETC...)	10

1. SCOPE AND APPLICATION

- 1.1. This method is applicable to the determination of Flashpoint by Pensky-Martens closed cup tester in a variety of wastes and liquids. It is based on ASTM D93-90 and SW846 Method 1010. Although the approximate working range is 20-200 °F, the test is generally considered complete when the temperature reaches 180°F without a measurable Flashpoint.
- 1.2. The associated LIMS method codes are AE (Method 1010) and GG (Method D93-90-Waste Matrix only).
- 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.

2. SUMMARY OF METHOD

- 2.1. The sample is heated at a slow constant rate with continual stirring if it is a liquid or water. A small test element is directed into the sample cup at regular intervals. The Flashpoint is the lowest temperature at which the test element causes the vapor above the sample to ignite.

3. DEFINITIONS

- 3.1. Refer to the glossary in the TestAmerica North Canton Laboratory Quality Manual (LQM), latest version.

4. INTERFERENCES

- 4.1. Not Applicable

5. SAFETY

- 5.1. Employees must abide by the policies and procedures in the Corporate Safety Manual, the facility addendum to the CSM, 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 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
p-Xylene	Flammable Irritant	100 ppm-TWA	Inhalation of vapors may be irritating to the nose and throat. Inhalation of high concentrations may result in nausea, vomiting, headache, ringing in the ears, and severe breathing difficulties, which may be delayed in onset. High vapor concentrations are anesthetic and central nervous system depressants. Skin contact results in loss of natural oils and often results in a characteristic dermatitis. May be absorbed through the skin. Vapors cause eye irritation. Splashes cause severe irritation, possible corneal 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. 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 when doing any other task that presents a strong possibility of getting cut. Disposable gloves that have been contaminated will be removed and discarded; other gloves will be cleaned immediately.
- 5.4. Exposure to chemicals must be maintained **as low as reasonably achievable** ; therefore, unless they are known to be non-hazardous, all samples must be opened, transferred and prepared in a fume hood, or under other means of mechanical ventilation, where possible. 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 will be kept closed unless transfers are being made.
- 5.5. The preparation of standards and reagents will be conducted in a fume hood with the sash closed as far as the operation will permit.
- 5.6. It is recommended that neat standards be purchased only as a last resort. The preparation of standards from neat materials and reagents should be conducted in a fume hood with the sash closed as far as the operations will permit.
- 5.7. Standards in solution may be diluted in the open laboratory when syringes and the like are utilized.

- 5.8. 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.
- 5.9. In the event a sample ignites in the test apparatus do not attempt to remove the sample. Turn off the apparatus and flame. The flame should go out when the cup is closed. If this does not happen the flame may be extinguished by covering the sample with a non-flammable material. After the apparatus has cooled the sample may be removed.
- 5.10. When testing a sample, the analyst shall remain within eyesight of the flash point tester and will manually shut down the tester if it fails to automatically shut down following ignition.

6. EQUIPMENT AND SUPPLIES

- 6.1. Ignitor and detector
- 6.2. Pensky-Martens closed cup tester with stirrer and stirring motor
- 6.3. Flash point sample cup
- 6.4. Thermometer: 20 - 200°F range (made for the Pensky-Martens) or Thermocouple
- 6.5. Barometer

7. REAGENTS AND STANDARDS

- 7.1. Reagents
 - 7.1.1. p-Xylene

8. SAMPLE COLLECTION, PRESERVATION AND STORAGE

- 8.1. Samples are not chemically preserved.
- 8.2. Samples are stored in glass containers at $4^{\circ}\text{C} \pm 2^{\circ}\text{C}$. Samples are not to be stored in plastic containers since volatile materials may diffuse through the walls of the enclosure.

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 and Method Blank) 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. Duplicates

9.2.1. 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.

9.2.2. Sample duplicates are performed at a minimum frequency of 10% per matrix (or one per analytical batch) and must meet laboratory-specific limits for precision. Soil and Waste matrices may be combined for batching purposes.

9.3. Control Limits

9.3.1. Control limits are established by the laboratory as described in SOP, NC-QA-0018.

9.3.2. Laboratory control limits are internally generated and updated periodically unless method specified. The latest version is easily accessible via the LIMs (QC Browser program).

9.4. Nonconformance and Corrective Action

9.4.1. 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. Calibration of tester

10.1.1. Determine the flash point of p-xylene following the procedures outlined in 11.4

10.1.2. The tester is operating properly when a value of $81 \pm 2^{\circ}\text{F}$ is obtained.

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 shall be completely documented using a Non-Conformance Memo and is approved by a Technical Specialist and QA Manager. The Non-Conformance Memo shall 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.

11.3. Sample Preparation Procedure

11.3.1. Not Applicable

11.4. Sample Analysis Procedure

11.4.1. Operate the Pensky-Martens tester according to the manufacturer's specifications, in a well-ventilated area away from flammable materials and significant air movement. Operating manuals are located in the laboratory near the tester. Performing this analysis under a hood is the best approach - ensure that the air intake and hood lights are turned off. Fill the sample cup to the designated line with sample and assemble the tester as directed.

11.4.1.1. Record the barometric pressure in cm.

11.4.2. A flash check must be analyzed with each analytical batch of samples. The compound p-Xylene is used to provide the analyst a reference true flash. The flash temperature is recorded on the analytical logsheet. The true flash temperature for p-Xylene is 81°F.

11.4.3. Pour the sample into the cup and record the initial temperature. If sample flashes, record the temperature at which the flash occurred on the analytical logsheet. Repeat sample for confirmation. If the temperature reaches 180°F and no flash occurs (see manual), turn off flashpoint and record > 180°F as the Flashpoint on the analytical logsheet.

11.4.4. If solvents are used to clean sample cups, be sure to clean thoroughly with reagent water to prevent contamination.

NOTE: Some samples may burn, but not flash. Record the initial temperature at which it burns and reanalyze for confirmation. Note this as a footnote when reporting.

Some samples have a Flashpoint below room temperature. If this is known, the sample should be chilled to just above freezing and then analyzed promptly to confirm a true Flashpoint.

11.5. Analytical Documentation

11.5.1. Record all analytical information in the analytical logsheet, which may be in electronic format, including the analytical data from standards, 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

12.1. If the sample did not flash, report > 180°F

12.2. Use the following equation to calculate the flashpoint:

$$\text{Flash} = F + [0.6 (76 - (BP \times 2.54))]$$

Where: F = Observed Flash

BP = Barometric Pressure in cm

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 that 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. This method does not contain any specific modifications that serve to minimize or prevent pollution.

15. WASTE MANAGEMENT

15.1. Refer to the Laboratory Sample and Waste Disposal plan.

- 15.2. Laboratory personnel assigned to perform hazardous waste disposal procedures must have a working knowledge of the established procedures and practices of TestAmerica North Canton. They must have training on the hazardous waste disposal practices upon initial assignment to these tasks, followed by an annual refresher training.
- 15.3. 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 Safety Manual for "Waste Management and Pollution Prevention."
- 15.4. Waste Streams Produced by the method
 - 15.4.1. The following waste streams are produced when this method is carried out.
 - 15.4.1.1. **Solid samples.** Solids are put into the red can for the debris waste stream
 - 15.4.1.2. **Liquid samples and waste solvents** Flammable wastes including the xylene standard are disposed of in the solvent waste stream located in the red can in the hood. Adding water to the solvent waste stream should be avoided.

16. REFERENCES

- 16.1. References
 - 16.1.1. Annual Book of ASTM Standards, ASTM Method D93-90, Flashpoint by Pensky-Martens Closed Tester.
 - 16.1.2. SW846, Test Methods for Evaluating Solid Waste, Third Edition, Pensky-Martens Closed Cup Method for Determining Ignitability, Method 1010
 - 16.1.3. Corporate Quality Management Plan (QMP), current version.
 - 16.1.4. TestAmerica Laboratory Quality Manual (LQM), current version.
- 16.2. Associated SOPs and Policies, latest version
 - 16.2.1. QA Policy, QA-003
 - 16.2.2. Glassware Washing, NC-QA-0014
 - 16.2.3. Statistical Evaluation of Data and Development of Control Charts, NC-QA-0018
 - 16.2.4. Supplemental Practices for DoD Project Work, NC-QA-0016

16.2.5. Standards and Reagents, NC-QA-0017

17. MISCELLANEOUS (TABLES, APPENDICES, ETC.)

17.1. Not Applicable

**Title: SPECIFIC GRAVITY BY HYDROMETER METHOD,
INSULATING OIL, API GRAVITY, AND FUEL DENSITY
[ASTM D1298-80]**

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This SOP was previously identified as SOP No. NCAP-11007.0.

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1.0 SCOPE AND APPLICATION

- 1.1 This method covers the laboratory determination, using a glass hydrometer, of the specific gravity and API gravity of petroleum products having a Reid Vapor Pressure (ASTM Standards D323) of 26 lbs. or less.
- 1.2 Specific gravity and API gravity are factors governing the quality of petroleum products. However, this is an uncertain indication of quality unless correlated with other properties.
- 1.3 Fuel Density is determined for use in the Heat of Combustion analysis for Liquid Hydrocarbons.

2.0 SUMMARY OF METHOD

- 2.1 The appropriate hydrometer is lowered into the sample and allowed to settle. After equilibrium has been reached, the hydrometer scale is read, and the temperature of the sample is noted.
- 2.2 Fuel Density is obtained from specific gravity values using Table 3 of the Petroleum Measurement Tables.

3.0 SAMPLE HANDLING

- 3.1 The samples are shielded from light until tested.
- 3.2 Temperature of sample - The density, relative density (specific gravity), or API gravity by the hydrometer method is most accurate at or near the reference temperature of 15°C or 60°F.

4.0 APPARATUS

- 4.1 Hydrometers, glass, graduated in units of specific gravity or API gravity as applicable (ASTM Standards D1298).
- 4.2 Thermometers - ASTM Standards D1298.
- 4.3 Hydrometer Cylinder, clear glass. For convenience in pouring, the cylinder may have a lip on the rim. The height of the cylinder should allow the appropriate hydrometer to float in the sample with at least 25mm clearance between the bottom of the hydrometer and the bottom of the cylinder.

4.3.1 For API gravity measurements on diesel fuel, use 250mL graduated cylinder.

5.0 SAFETY

- 5.1 During the execution of the procedures outlined in this SOP, potentially hazardous compounds may be encountered. Personnel should observe all appropriate safety rules as outlined in the TestAmerica Safety Manual.
- 5.2 Samples handled under this SOP may originate from unknown sites, which could contain a variety of hazardous materials. Appropriate safety rules as outline in the TestAmerica Safety Manual should be observed while handling samples of unknown content and/or origin.

6.0 PROCEDURE

- 6.1 Transfer the sample to clean hydrometer cylinder without splashing, to avoid the formation of air bubbles, and to reduce to a minimum evaporation of the lower boiling constituents of more volatile samples. Remove any air bubbles formed, after they have collected on the surface of the sample, by touching them with a piece of clean filter paper or lightly tapping the cylinder before inserting the hydrometer.
- 6.1.1 Approximate Sample Volume - 100mL for transformer oils or 250mL for Diesel fuel.
- 6.2 Place the cylinder containing the sample in a vertical position in a location free from air currents. Ensure that the temperature of the sample does not change appreciably during the time necessary to complete the test; during this period, the temperature of the surrounding medium should not change more than 2°C (5°F).
- 6.2.1 Record temperature to the nearest 1.0°F.
- 6.3 Lower the hydrometer gently into the sample. Take care to avoid wetting the stem above the level to which it will be immersed in the liquid. Depress the hydrometer about two scale divisions into the liquid, and then release it. The remainder of the stem of the hydrometer, which is above the level of the liquid, must be kept dry since unnecessary liquid on the stem affects the reading obtained. With samples of low viscosity, impart a slight spin to the hydrometer on releasing to assist in bringing it to rest, floating freely away from the walls of the cylinder. Allow sufficient time for the hydrometer to come to rest, and for all air bubbles to come to the surface. This is particularly necessary in the case of more viscous samples.

- 6.4 When the hydrometer has come to rest, floating freely away from the walls of the cylinder, estimate the hydrometer scale reading to the nearest 0.001 for specific gravity or 0.1° API. The correct hydrometer reading is that point on the hydrometer scale at which the principal surface of the liquid cuts the scale. Determine this point by placing the eye slightly below the level of the liquid and slowly raising it until the surface, first seen as a distorted ellipse, appears to become a straight line cutting the hydrometer scale.

- 6.5 Opaque liquid - See ASTM D1298.

7.0 POLLUTION PREVENTION

- 7.1 All wastes from this procedure shall be disposed following the recommendations of the TestAmerica Safety Manual.

8.0 CALCULATIONS AND REPORTS

- 8.1 When a relative density (specific gravity) hydrometer has been employed, use Tables 23 from the Petroleum Measurement Tables (see D1250), to obtain Relative Density (Specific Gravity) 60/60°F.
- 8.2 When an API hydrometer has been employed, use Petroleum Measurement Tables 5 to reduce the observed API Gravity to 60°F. Use Table 3 to convert the API Gravity at 60°F to Specific Gravity 60/60°F. Fuel Density for SOP 0370 can be calculated from Specific Gravity 60/60°F by converting Table 3 values to pounds per gallon (multiply kg/L x 2.2 x 3.785).
- 8.3 Report the final value as relative density (specific gravity) at 60/60°F to 0.001 and/or API gravity 60°F to the nearest 0.1.

9.0 REFERENCES

- 9.1 D323 Test Method for Vapor Pressure of Petroleum Products (Reid Method).¹
- 9.2 D1250 Petroleum Measurement Tables.¹
- 9.3 D1298-80 Standard Test Method for Density, Relative Density (specific gravity), or D-287 API Gravity of Crude Petroleum and Liquid Petroleum Products by Hydrometer Method.¹
- 9.4 E1 Specification for ASTM Thermometers.¹

¹Annual Book of ASTM Standards, Vol 05.01.

9.5 TestAmerica Quality Assurance Manual, current revision.

9.6 TestAmerica Safety Manual, current revision.

10.0 METHOD MODIFICATIONS

None

11.0 REVISION HISTORY

Revision 1, dated 2/15/08

- Reformatted to TestAmerica style and content.

¹Annual Book of ASTM Standards, Vol. 05.03 and 14.01.

Title: KINEMATIC VISCOSITY OF LIQUID PETROLEUM PRODUCTS (AT 100°F, 210°F, 40°C, 100°C), AND CONVERSION OF KINEMATIC VISCOSITY cST) TO SAYBOLT UNIVERSAL VISCOSITY (SUS)
[ASTM D445, D2161]

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1.0 SCOPE AND APPLICATION

- 1.1 Viscosity influences properties of petroleum products such as heat transfer properties, pressure drop, oil flow, speed of moving parts, and insulating oil processing conditions.

2.0 SUMMARY OF METHOD

- 2.1 The time is measured in seconds for a fixed volume of liquid to flow under gravity through the capillary of a calibrated viscometer under a reproducible driving head and at a closely controlled temperature. The kinematic viscosity is the product of the measured flow time and the calibration constant of the viscometer.

2.1.1

Temperature of Bath	Units Reported
100°F	SUS
210°F	SUS
40°C	CST
100°C	CST

3.0 INTERFERENCES

- 3.1 The viscosity of steam-refined cylinder oils, black lubricating oils, residual fuel oils and similar waxy products can be affected by the previous thermal history.

4.0 APPARATUS¹

- 4.1 Precision Science Temp-Trol Viscosity Bath or equivalent.
- 4.2 Philadelphia Micro-set Rheostat or equivalent.
- 4.3 Cannon-Ubbelohde Viscometers (Calibrated) or equivalent suspended-level type viscometers.

¹See ASTM D445, Annual Book of ASTM Standards, Vol. 05.01.

4.4 Thermometers (ASTM 28F, ASTM 30F, ASTM 120C, ASTM 121C) or equivalents.

4.5 Stop watch or equivalent.

5.0 REAGENTS

5.1 Iso-Octane

5.2 Acetone.

6.0 SAFETY

6.1 During the execution of the procedures outlined in this SOP, potentially hazardous compounds may be encountered. Personnel should observe all appropriate safety rules as outlined in the Chemical Hygiene Plan.

6.2 Samples handled under this SOP may originate from unknown sites, which could contain a variety of hazardous materials. Appropriate safety rules as outline in the Chemical Hygiene Plan should be observed while handling samples of unknown content and/or origin.

7.1 PROCEDURE

7.1 Turn on viscosity bath and preheat; use cooling water as necessary to maintain the required test temperature.

7.1.1 The temperature of viscosity bath used is determined by the manufacturer's recommendation, customer's requirements, and/or previous history of sample being tested.

7.1.2 Record the temperature of the bath used.

7.2 Select a clean, dry, calibrated viscometer having a range covering the estimated viscosity (for example, a wide capillary for a very viscous liquid and a narrower capillary for a more fluid liquid).

7.2.1 Check for a past history of tubes used on the sample.

7.2.2 For very thick fluids, take care that measurements are made at sufficiently high temperatures for such materials to flow freely so that similar results will be obtained in viscometers of different capillary diameters.

7.2.3 Record Viscometer Number and corresponding constant used.

7.3 Fill the viscometer with the sample to be tested.

- 7.3.1 Invert sample and swirl before filling to ensure homogeneity.
- 7.3.2 Introduce sample through tube G into the lower reservoir, adding enough sample to bring the level between lines J and K (see 10.1).
- 7.4 Place the viscometer into the holder and insert it into the bath.
 - 7.4.1 More than 1 viscometer can be placed in the bath at the same time. Never add or withdraw a viscometer while any other viscometer in the bath is in use for measuring a flow time.
- 7.5 Allow ~20 minutes for sample to reach bath temperature.
 - 7.5.1 Adjust the volume of the sample after it has reached temperature equilibrium if needed.
- 7.6 Place a finger over tube B and apply pressure (with a bulb) to tube G until sample reaches the top of tube A. Remove bulb and finger to allow liquid to start draining from tube A.
- 7.7 To measure the afflux time, measure the time for the meniscus of the sample to pass Mark D to Mark F to the nearest 0.1 second.
 - 7.7.1 The afflux time can not be less than 200 seconds. If it is, select a viscometer with a capillary of a smaller diameter and repeat procedure.
 - 7.7.2 During this time, the temperature of the bath should not move from the lined temperature increment at which the bath is set (See 2.1.1).
 - 7.7.2.1 Deviation: If bath temperature control is inadequate to maintain temperature at the exact temperature required, 0.1°F must be maintained for the 100°F bath and the 212°F (100°C) bath and 0.1°C for the 40°C bath. The potential error associated with 0.1 degree is 0.1% for diesel fuels at 100°F, 0.15% for lubricating oils at 40°C and 2% for lubricants at 100°C.
- 7.8 Repeat 6.6 - 6.7 until 2 measurements of afflux time agree within 0.2% and record.
- 7.9 To clean - rinse with Iso-Octane until clean, rinse with acetone, blow dry with compressed air, and let dry in 50°C oven for 2 minutes.
- 7.10 Viscometer should be calibrated once a year.

8.0 CALCULATIONS

8.1 Kinematic Viscosity (40°C and 100°C):

$V = Ct$; V = kinematic viscosity, cSt.

C = calibration constant of viscometer, cSt/s.

t = efflux time, s.

8.1.1 Report the average of the two values to the tenth.

8.2 Saybolt Universal Viscosity (100°F and 210°F):

Convert Kinematic Viscosity (CST) to Saybolt Universal Viscosity (SUS) and report the average of the two values to the unit.

8.2.1 Use Table 1 in ASTM D2161².

8.2.1.1 Obtain viscosities not listed, but which are within the range given in Table 1, by linear interpolation.

8.3 Precision:

8.3.1 *Repeatability* - exceed 0.35% of their mean only in one case in twenty.

8.3.2 *Reproducibility* - exceed 0.70% of their mean only in one case in twenty.

9.0 REFERENCES

9.1 ASTM Standards:

9.1.1 D117 Standard Guide to Test Methods and Specifications for Electrical Insulating Oils of Petroleum Origin.³

9.1.2 D445 Standard Test Method for Kinematic Viscosity of Transparent and Opaque Liquids (and the Calculation of Dynamic Viscosity).⁴

9.1.3 D446 Standard Specifications and Operating.⁴

²1990 Annual Book of ASTM Standards, Vol.05.02.

³1990 Annual Book of ASTM Standards, Vol. 10.03.

⁴1990 Annual Book of ASTM Standards, Vol. 05.01.

9.1.4D2161 Standard Practice for Conversion of Kinematic Viscosity of Saybolt
Universal Viscosity or to Saybolt Furol Viscosity.⁵

9.2 TestAmerica Quality Assurance Manual, current revision.

9.3 TestAmerica Safety Manual, current revision.

10.0 METHOD MODIFICATIONS

None

11.0 REVISION HISTORY

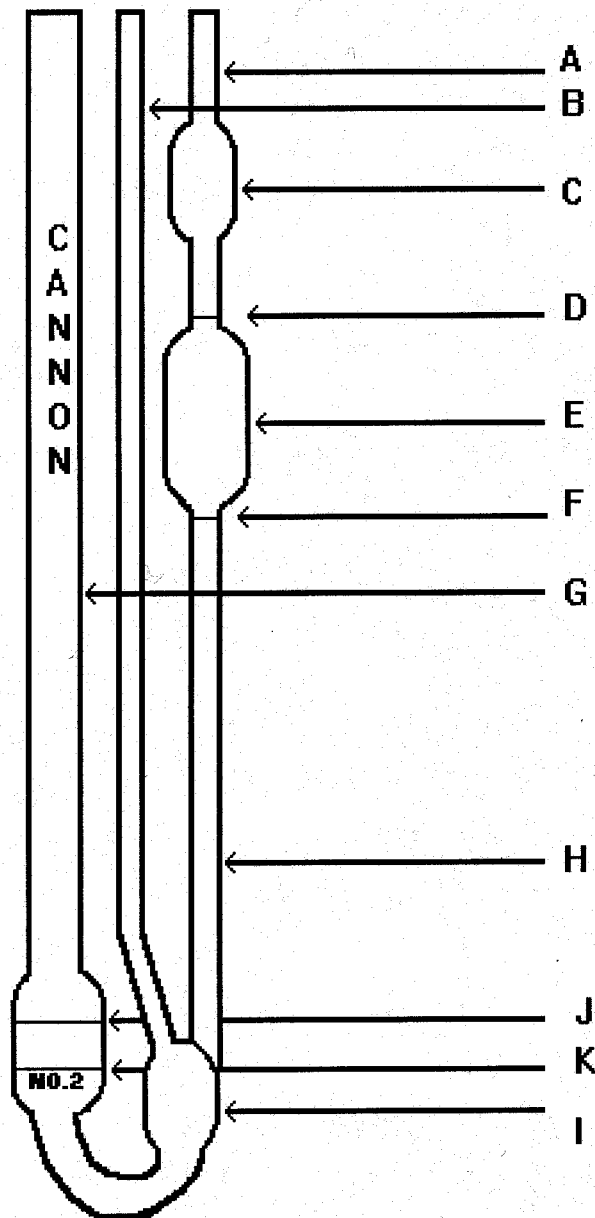
Revision 1, dated 2/28/08

- Reviewed and reformatted to TestAmerica style and format.

⁵1990 Annual Book of ASTM Standards, Vol. 05.02.

10.0 TABLES

10.1 Cannon-Ubbelohde Viscometer



Cover Page:

Quality Assurance Manual

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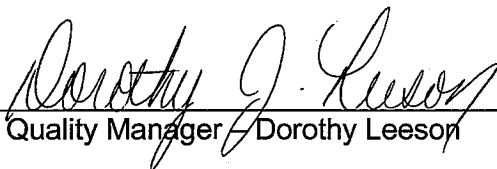
**Quality Assurance Manual
Approval Signatures**



Laboratory Director – Opal Davis-Johnson

12/10/07

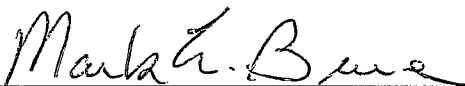
Date



Quality Manager – Dorothy Leeson

12/10/07

Date



Technical Director – Dr. Mark Bruce

12/10/07

Date

SECTION 2

TABLE OF CONTENTS

Section No.	Title	Page No.	Effective Date
-	COVER PAGE	COVER	01/01/2008
1.0	TITLE PAGE	1-1	01/01/2008
2.0	TABLE OF CONTENTS	2-1	01/01/2008
3.0	INTRODUCTION	3-1	01/01/2008
3.1	Introduction And Compliance References	3-1	01/01/2008
3.2	Terms And Definitions	3-1	01/01/2008
3.3	Scope / Fields Of Testing	3-1	01/01/2008
3.4	Management Of The Manual	3-2	01/01/2008
4.0	ORGANIZATION AND MANAGEMENT (<i>NELAC 5.4.1</i>)	4-1	01/01/2008
4.1	Overview	4-1	01/01/2008
4.2	Roles And Responsibilities	4-2	01/01/2008
5.0	QUALITY SYSTEM (<i>NELAC 5.4.2</i>)	5-1	01/01/2008
5.1	Quality Policy Statement	5-1	01/01/2008
5.2	Ethics And Data Integrity	5-1	01/01/2008
5.3	Quality System Supporting Documentation	5-2	01/01/2008
5.4	Qa/Qc Objectives For The Measurement Of Data	5-3	01/01/2008
5.5	Criteria For Quality Indicators	5-5	01/01/2008
5.6	Statistical Quality Control	5-5	01/01/2008
5.7	Quality System Metrics	5-6	01/01/2008
6.0	DOCUMENT CONTROL (<i>NELAC 5.4.3</i>)	6-1	01/01/2008
6.1	Overview	6-1	01/01/2008
6.2	Document Approval And Issue	6-1	01/01/2008
6.3	Procedures For Document Control Policy	6-2	01/01/2008
6.4	Obsolete Documents	6-2	01/01/2008
7.0	REVIEW OF WORK REQUEST	7-1	01/01/2008
7.1	Overview	7-1	01/01/2008
7.2	Review Sequence And Key Personnel	7-2	01/01/2008
7.3	Documentation	7-3	01/01/2008
8.0	SUBCONTRACTING OF TESTS (<i>NELAC 5.4.5</i>)	8-1	01/01/2008
8.1	Overview	8-1	01/01/2008
8.2	Qualifying And Monitoring Subcontractors	8-2	01/01/2008
8.3	Oversight And Reporting	8-5	01/01/2008
8.4	Contingency Planning	8-6	01/01/2008
9.0	PURCHASING SERVICES AND SUPPLIES (<i>NELAC 5.4.6</i>)	9-1	01/01/2008
9.1	Overview	9-1	01/01/2008

Section No.	Title	Page No.	Effective Date
9.2	Glassware	9-1	01/01/2008
9.3	Reagents, Standards & Supplies	9-1	01/01/2008
9.4	Purchase Of Equipment/Instruments/Software	9-3	01/01/2008
9.5	Services	9-4	01/01/2008
9.6	Suppliers	9-4	01/01/2008
10.0	SERVICE TO THE CLIENT (<i>NELAC 5.4.7</i>)	10-1	01/01/2008
10.1	Overview	10-1	01/01/2008
10.2	Special Services	10-1	01/01/2008
10.3	Client Communication	10-1	01/01/2008
10.4	Reporting	10-1	01/01/2008
10.5	Client Surveys	10-2	01/01/2008
11.0	COMPLAINTS (<i>NELAC 5.4.8</i>)	11-1	01/01/2008
11.1	Overview	11-1	01/01/2008
11.2	External Complaints	11-1	01/01/2008
11.3	Internal Complaints	11-2	01/01/2008
11.4	Management Review	11-2	01/01/2008
12.0	CONTROL OF NON-CONFORMING WORK (<i>NELAC 5.4.9</i>)	12-1	01/01/2008
12.1	Overview	12-1	01/01/2008
12.2	Responsibilities And Authorities	12-1	01/01/2008
12.3	Evaluation Of Significance And Actions Taken	12-2	01/01/2008
12.4	Prevention Of Nonconforming Work	12-2	01/01/2008
12.5	Method Suspension/Restriction (Stop Work Procedures)	12-2	01/01/2008
13.0	CORRECTIVE ACTION (<i>NELAC 5.4.10</i>)	13-1	01/01/2008
13.1	Overview	13-1	01/01/2008
13.2	Definitions	13-1	01/01/2008
13.3	General	13-1	01/01/2008
13.4	Closed Loop Corrective Action Process	13-2	01/01/2008
13.5	Technical Corrective Actions	13-3	01/01/2008
13.6	Basic Corrections	13-3	01/01/2008
14.0	PREVENTIVE ACTION (<i>NELAC 5.4.11</i>)	14-1	01/01/2008
14.1	Overview	14-1	01/01/2008
14.2	Management Of Change	14-2	01/01/2008
15.0	CONTROL OF RECORDS (<i>NELAC 5.4.12</i>)	15-1	01/01/2008
15.1	Overview	15-1	01/01/2008
15.2	Technical And Analytical Records	15-5	01/01/2008
15.3	Laboratory Support Activities	15-6	01/01/2008
15.4	Administrative Records	15-6	01/01/2008
15.5	Records Management, Storage And Disposal	15-6	01/01/2008
16.0	AUDITS (<i>NELAC 5.4.13</i>)	16-1	01/01/2008
16.1	Overview	16-1	01/01/2008
16.2	Technical And Analytical Records	16-1	01/01/2008
16.3	External Audits	16-3	01/01/2008
16.4	Audit Findings	16-5	01/01/2008
17.0	MANAGEMENT REVIEWS (<i>NELAC 5.4.14</i>)	17-1	01/01/2008

Section No.	Title	Page No.	Effective Date
17.1	Quality Assurance Report	17-1	01/01/2008
17.2	Annual Management Review	17-2	01/01/2008
17.3	Potential Integrity Related Managerial Reviews	17-3	01/01/2008
18.0	PERSONNEL (NELAC 5.5.2)	18-1	01/01/2008
18.1	Overview	18-1	01/01/2008
18.2	Education And Experience Requirements For Technical Personnel	18-1	01/01/2008
18.3	Training	18-3	01/01/2008
18.4	Data Integrity And Ethics Training Program	18-4	01/01/2008
19.0	ACCOMMODATIONS AND ENVIRONMENTAL CONDITIONS (NELAC 5.5.3)	19-1	01/01/2008
19.1	Overview	19-1	01/01/2008
19.2	Environment	19-1	01/01/2008
19.3	Work Areas	19-2	01/01/2008
19.4	Floor Plan	19-2	01/01/2008
19.5	Building Security	19-2	01/01/2008
20.0	TEST METHODS AND METHOD VALIDATION (NELAC 5.5.4)	20-1	01/01/2008
20.1	Overview	20-1	01/01/2008
20.2	STANDARD OPERATING PROCEDURES (Sops)	20-1	01/01/2008
20.3	Laboratory Methods Manual	20-1	01/01/2008
20.4	Selection Of Methods	20-2	01/01/2008
20.5	Laboratory Developed Methods And Non-Standard Methods	20-16	01/01/2008
20.6	Validation Of Methods	20-16	01/01/2008
20.7	Method Detection Limits (Mdl)/ Limits Of Detection (Lod)	20-18	01/01/2008
20.8	Instrument Detection Limits (Idl)	20-19	01/01/2008
20.9	Verification Of Detection And Reporting Limits	20-20	01/01/2008
20.10	Retention Time Windows	20-20	01/01/2008
20.11	Evaluation Of Selectivity	20-21	01/01/2008
20.12	Estimation Of Uncertainty Of Measurement	20-21	01/01/2008
20.13	Control Of Data	20-22	01/01/2008
21.0	EQUIPMENT (AND CALIBRATIONS) (NELAC 5.5.5)	21-1	01/01/2008
21.1	Overview	21-1	01/01/2008
21.2	Preventive Maintenance	21-1	01/01/2008
21.3	Support Equipment	21-3	01/01/2008
21.4	Instrument Calibrations	21-5	01/01/2008
21.5	Policy On Tentatively Identified Compounds (Tics) – Gc/Ms Analysis	21-13	01/01/2008
21.6	Policy On Gc/Ms Tuning	21-13	01/01/2008
22.0	MEASUREMENT TRACEABILITY (NELAC 5.5.6)	22-1	01/01/2008
22.1	Overview	22-1	01/01/2008
22.2	Nist-Traceable Weights And Thermometers	22-2	01/01/2008
22.3	Reference Standards / Materials	22-2	01/01/2008

Section No.	Title	Page No.	Effective Date
22.4	Documentation And Labeling Of Standards, Reagents, And Reference Materials	22-3	01/01/2008
23.0	SAMPLING (<i>NELAC 5.5.7</i>)	23-1	01/01/2008
23.1	Overview	23-1	01/01/2008
23.2	Sampling Containers	23-1	01/01/2008
23.3	Field Quality Control (Qc)	23-1	01/01/2008
23.4	Definition Of Holding Time	23-2	01/01/2008
23.5	Sampling Containers, Preservation Requirements, Holding Times	23-3	01/01/2008
23.6	Sample Aliquots / Subsampling	23-3	01/01/2008
24.0	HANDLING OF SAMPLES (<i>NELAC 5.5.8</i>)	24-1	01/01/2008
24.1	Chain Of Custody (Coc)	24-1	01/01/2008
24.2	Sample Receipt	24-2	01/01/2008
24.3	Sample Acceptance Policy	24-4	01/01/2008
24.4	Sample Storage	24-4	01/01/2008
24.5	Hazardous Samples And Foreign Soils	24-5	01/01/2008
24.6	Sample Shipping	24-5	01/01/2008
24.7	Sample Disposal	24-5	01/01/2008
25.0	ASSURING THE QUALITY OF TEST RESULTS (<i>NELAC 5.5.9</i>)	25-1	01/01/2008
25.1	Overview	25-1	01/01/2008
25.2	Controls	25-1	01/01/2008
25.3	Negative Controls	25-1	01/01/2008
25.4	Positive Controls	25-2	01/01/2008
25.5	Sample Matrix Controls	25-4	01/01/2008
25.6	Acceptance Criteria (Control Limits)	25-6	01/01/2008
25.7	METHOD DETECTION LIMITS (Mdls)	25-8	01/01/2008
25.8	Additional Procedures To Assure Quality Control	25-8	01/01/2008
26.0	REPORTING RESULTS (<i>NELAC 5.5.10</i>)	26-1	01/01/2008
26.1	Overview	26-1	01/01/2008
26.2	Test Reports	26-1	01/01/2008
26.3	Reporting Level Or Report Type	26-3	01/01/2008
26.4	Supplemental Information For Test	26-4	01/01/2008
26.5	Environmental Testing Obtained From Subcontractors	26-4	01/01/2008
26.6	Client Confidentiality	26-4	01/01/2008
26.7	Format Of Reports	26-5	01/01/2008
26.8	Amendments To Test Reports	26-5	01/01/2008
26.9	Policies On Client Requests For Amendments	26-5	01/01/2008

LIST OF TABLES

Table No.	Title	Page	Effective Date
9-1	<u>Storage of Reagents and Chemicals</u>	9-6	01/01/2008
13-1	<u>Example - General Corrective Action Procedures</u>	13-5	01/01/2008
15-1	<u>Records and Retention Schedule</u>	15-1	01/01/2008
15-2	<u>Special Record Retention Requirements</u>	15-3	01/01/2008
16-1	<u>Audit Types and Frequency</u>	16-1	01/01/2008
20-1	<u>Wet Chemistry Methods</u>	20-3	01/01/2008
20-2	<u>Methods for Mercury by Cold Vapor Atomic Absorption</u>	20-7	01/01/2008
20-3	<u>Methods for Mercury by Cold Vapor Atomic Fluorescence</u>	20-7	01/01/2008
20-4	<u>Methods for Metals by ICP and ICPMS</u>	20-7	01/01/2008
20-5	<u>Metals Sample Preparation Methods</u>	20-11	01/01/2008
20-6	<u>Organic Sample Preparation Methods</u>	20-12	01/01/2008
20-7	<u>Organic Method of Analysis</u>	20-14	01/01/2008
21-1	<u>Example - Laboratory Equipment & Instrumentation</u>	21-15	01/01/2008
21-2	<u>Example – Laboratory Balance Inventory</u>	21-19	01/01/2008
21-3	<u>Example – Laboratory Refrigerators, Freezers, Ovens, Thermometers, Steambaths, Hotblocks, and Hotplates</u>	21-20	01/01/2008
21-4	<u>Example: Schedule of Routine Maintenance</u>	21-22	01/01/2008
21-5	<u>Example: Periodic Equipment Calibrations</u>	21-32	01/01/2008
21-6	<u>Example: Preventive Maintenance Procedures</u>	21-33	01/01/2008

Table No.	Title	Page	Effective Date
23-1	<u>Inorganic Sample Containers, Preservatives, and Holding Times</u>	23-5	01/01/2008
23-2	<u>Organic Sample Containers, Preservatives, and Holding Times</u>	23-15	01/01/2008
23-3	<u>Sample Containers, Preservatives, and Holding Times for TCLP and SPLP</u>	23-23	01/01/2008

LIST OF FIGURES

Figure No.	Title	Page	Effective Date
3-1	<u>Example - Format for a QA/QC Policy Memorandum</u>	3-4	01/01/2008
4-1	<u>Corporate Organizational Chart</u>	4-9	01/01/2008
8-1	<u>Example - Client-Approved Subcontractor Form</u>	8-7	01/01/2008
8-2	<u>Example - Subcontracting Laboratory Approval Form (Initial / Renewal)</u>	8-8	01/01/2008
9-2	<u>Example - JD Edwards Vendor Add Request Form</u>	9-7	01/01/2008
16-1	<u>Example - Internal Audit Workbook</u>	16-7	01/01/2008
16-2	<u>Example – Internal Audit System Checklist</u>	16-8	01/01/2008
17-1	<u>Example - QA Monthly Report to Management</u>	17-4	01/01/2008
17-2	<u>Example – Laboratory Metrics Categories</u>	17-6	01/01/2008
20-1	<u>Example – Demonstration of Capability Documentation</u>	20-31	01/01/2008
20-2	<u>Example – New Method / Additional Analyte Checklist</u>	20-32	01/01/2008
20-3	<u>Work Flow</u>	20-33	01/01/2008
24-1	<u>Example: Chain of Custody (COC)</u>	24-7	01/01/2008
24-2	<u>Example: Custody Seals</u>	24-8	01/01/2008
24-3	<u>Example: Internal Chain of Custody (COC)</u>	24-9	01/01/2008
24-4	<u>Example: Cooler Receipt Form</u>	24-11	01/01/2008

LIST OF APPENDICES

Appendix No.	Title	Page	Effective Date
1	<u>TestAmerica Ethics Policy No. CA-L-P-001</u>	Appendix 1-1	01/01/2008
2	<u>Example - Laboratory Organization Chart</u>	Appendix 2-1	01/01/2008
3	<u>Laboratory Floor Plan</u>	Appendix 3-1	01/01/2008
4	<u>Laboratory Method Listing</u>	Appendix 4-1	01/01/2008
5	<u>Laboratory Reporting Limits</u>	Appendix 5-1	01/01/2008
6	<u>Glossary/Acronyms</u>	Appendix 6-1	01/01/2008
7	<u>Laboratory Certifications, Accreditations, Validations</u>	Appendix 7-1	01/01/2008
8	<u>Data Qualifiers</u>	Appendix 8-1	01/01/2008

SOPs AND POLICIES REFERRED TO IN THE QA MANUAL

SOP/Policy Reference	Title
CA-C-S-001	Work Sharing Process
CA-L-S-001	Internal Investigation of Potential Data Discrepancies and Determination for Data Recall
CA-L-P-001	Ethics Policy
CA-L-P-002	Contract Compliance Policy
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-005	Calibration Curves (General)
CA-T-P-001	Qualified Products List
CORP-MS-0001NC	GC/MS Analysis Based on Method 8270C,
CORP-MS-0002NC	Determination of Volatile Organics by GC/MS based on Method 8260B & 8260A).
CORP-QA-0010	Nonconformance and Corrective Action System
CORP-QA-0013	Employee Orientation and Training
CW-F-P-002	Authorization Matrix
CW-F-S-004	Controlled Purchases Policy
CW-L-P-001	Record Retention
CW-Q-S-001	Corporate Document Control and Archiving
CW-Q-S-002	Writing a Standard Operating Procedure (SOPs)
NC-QA-0012	Shipping Department
NC-QA-015	Equipment Monitoring and Thermometer Calibration
NC-QA-0017	Standards and Reagents
NC-QA-0018	Statistical Evaluation of Data and Development of Control Charts
NC-QA-0019	Records Information Management
NC-QA-0020	Laboratory Holding Blanks
NC-QA-0021	Evaluation of Method Detection Limits for Chemical Tests

NC-QA-0027	Preparation and Management of Standard Operating Procedures
NC-SC-0005	Sample Receiving and Sample Control
NC-SC-0006	Sample Procurement Protocol
QA-003	TestAmerica North Canton Quality Control Program
S-C-002	Complaint Handling and Service Recovery
S-Q-001	Document Control
S-Q-004	Acceptable Manual Integration Practices

SECTION 3

INTRODUCTION (NELAC 5.1 - 5.3)

3.1 INTRODUCTION AND COMPLIANCE REFERENCES

The TestAmerica North Canton Quality Assurance Manual (QAM) is a document prepared to define the overall policies, organization objectives and functional responsibilities for achieving TestAmerica 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 (1999). In addition, the policies and procedures outlined in this manual are compliant with the various accreditation and certification programs listed in Appendix 7. 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.
- EPA SW-846, *Test Methods for the Evaluation of Solid Waste*, 3rd Edition, September 1986; Update I, July 1992; Update II, September 1994; and Update III, December 1996.
- 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 that data produced by TestAmerica North Canton 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 for the Glossary/Acronyms.

3.3 SCOPE / FIELDS OF TESTING

TestAmerica analyzes thousands 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 water, 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 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 TestAmerica North Canton 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 will abide by the requested criteria following review and acceptance of the requirements by the Laboratory Director, 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

The manual is reviewed annually by the QA Manager and laboratory personnel to assure that it reflects current practices and meets the requirements of TestAmerica North Canton clients and regulators. Occasionally, the manual may need changes in order to meet new or changing regulations and operations. The QA Manager will review the changes in the normal course of business and incorporate changes into revised sections of the document. The updates will be reviewed by the QA Manager, Laboratory Director/Manager, Technical Director(s), relevant operational staff and Corporate Quality Assurance (if a change is made to the Corporate template) and then formally incorporated into the document in periodic updates. The QAM is based on a Corporate QAM Template that is prepared and approved by the Chief Operating Officers (COOs) and Corporate Quality Assurance. This template is reviewed annually by the COOs, Corporate Quality, and each laboratory. Necessary changes are coordinated by the Vice President of Quality and Environmental Health & Safety (EHS) and distributed to each laboratory for inclusion in the laboratory specific QA Manuals.

Policies in the QAM that require immediate attention may be addressed through the use of Corporate 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. In addition, Corporate QA/QC Policy

Memoranda are signed by the COOs and VP of Quality and EHS. 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. A similar procedure is followed for local laboratory changes.

3.4.2 Control

This manual is considered confidential within TestAmerica and may not be altered in any manner by other than a duly appointed representative from TestAmerica. If the document has been provided to external users or regulators, it is for the exclusive purpose of reviewing TestAmerica North Canton quality systems and shall not be used in any other way without the written permission of an appointed representative of TestAmerica. The procedure for control of distribution is incorporated by reference to SOP S-Q-001, Document Control.

The order of precedence in the event of a conflict between policies is outlined in Section 5.3 of this Quality Assurance Manual.

Figure 3-1.

Example - Format for a QA/QC Policy Memorandum

TestAmerica North Canton	
SOP/LQM CHANGE FORM	
SOP/LQM NUMBER:	
TITLE:	
SECTION(S) AFFECTED BY CHANGE:	
REASON FOR ADDITION OR CHANGE:	
CHANGE EFFECTIVE FROM: (DATE):	
CHANGE OR ADDITION:	
SUBMITTED BY/DATE:	
*APPROVED BY:	
Technical Reviewer Signature	Date
Environmental Health & Safety Signature	Date
QA Signature	Date
Technical Director (if applicable)	Date
Laboratory Director Signature	Date

SECTION 4

ORGANIZATION AND MANAGEMENT (NELAC 5.4.1)

4.1 OVERVIEW

TestAmerica North Canton is part of a national network of laboratories known as TestAmerica. This Quality Assurance Manual (QAM) is applicable to the TestAmerica North Canton laboratory only.

TestAmerica North Canton Laboratory
4101 Shuffle Drive NW
North Canton, OH 44720
EPA I.D. Code No. 9503

The Corporate organization chart can be found in Figure 4-1 and the laboratory organization chart can be found in Appendix 2. The locations of other TestAmerica labs are as follows:

Aerotech Environmental Laboratories (AEL)
TestAmerica Anchorage
TestAmerica Austin
TestAmerica Buffalo
TestAmerica Buffalo Grove
TestAmerica Burlington
TestAmerica Cedar Falls
TestAmerica Chicago
TestAmerica Colorado Springs
TestAmerica Connecticut
TestAmerica Corpus Christi
TestAmerica Dayton
TestAmerica Denver
TestAmerica Edison
TestAmerica Honolulu
TestAmerica Houston
TestAmerica Irvine
TestAmerica King of Prussia
TestAmerica Knoxville
TestAmerica Los Angeles
TestAmerica Mobile
TestAmerica Morgan Hill
TestAmerica Nashville
TestAmerica New Orleans
TestAmerica Ontario
TestAmerica Orlando
TestAmerica Pensacola
TestAmerica Phoenix
TestAmerica Pittsburgh

TestAmerica Portland
TestAmerica Richland
TestAmerica San Francisco
TestAmerica Savannah
TestAmerica Seattle
TestAmerica Spokane
TestAmerica St. Louis
TestAmerica Tacoma
TestAmerica Tallahassee
TestAmerica Tampa
TestAmerica Valparaiso
TestAmerica Watertown
TestAmerica West Sacramento
TestAmerica Westfield

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. More extensive job descriptions are maintained by laboratory management.

4.2.1 Quality Assurance Program

The responsibility for quality lies with every employee of TestAmerica North Canton. All employees have access to the QAM 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.

4.2.2 Chairman/Chief Executive Officer (CEO)

The Chairman/CEO is the Chairman of the Board of Directors and is ultimately responsible for the quality and performance of all TestAmerica facilities. Together with the President/CEO of the Analytical Division, the Chairman/CEO establishes the overall quality standard and data integrity program for the company, providing the necessary leadership and resources to assure that the standard and integrity program are met.

4.2.3 President/Chief Executive Officer (CEO)

The President/CEO is a member of the Board of Directors and is ultimately responsible for the quality and performance of all TestAmerica facilities. Together with the Chairman/CEO, the President/CEO establishes the overall quality standard and data integrity program for the Analytical Division, providing the necessary leadership and resources to assure that the standard and integrity program are met.

4.2.4 Chief Operating Officer (COO) – East and West

The COOs serve as the ranking executives for all respective analytical laboratory operational functions and report to the President/CEO of the Analytical Division. They are responsible for the daily management of all analytical laboratories, long-term planning and development of technical policies and management plans. They ensure the attainment of corporate objectives through the selection, development, motivation, and evaluation of top management personnel. The COOs approve all operating budgets and capital expenditures. The COOs sign-off on the final QAM template that contains company policies for implementing the Quality Program.

4.2.5 General Manager (GM)

Each GM reports directly to a COO. Each GM has full responsibility for the overall administrative and operational management of their respective laboratories. The GM's responsibilities include allocation of personnel and resources, long-term planning, setting goals, and achieving the financial, business, and quality objectives of TestAmerica. The GM ensures timely compliance with corporate management directives, policies, and management systems reviews. The GM is also responsible for restricting any laboratory from performing analyses that cannot be consistently and successfully performed to meet the standards set forth in this manual.

4.2.6 Vice President of Quality and Environmental Health and Safety (VP-QA/EHS)

The Vice President of QA/EHS reports directly to the Chairman/CEO. With the aid of the Analytical Division and Non-Analytical Division Senior Management Teams, Laboratory Director/Managers, Quality Directors, EHS Directors, QA Managers and EHS Coordinators, the VP-QA/EHS has the responsibility for the establishment, general overview and Corporate maintenance of the Quality Assurance and Environmental, Health and Safety Program within TestAmerica. Additional responsibilities include:

- Review of QA/QC aspects of Corporate SOPs, national projects and expansions or changes in services.
- Coordination/preparation of the Corporate QAM Template that is used by each laboratory to prepare its own laboratory-specific QAM.
- Maintenance of Corporate Policies, Quality Memorandums and SOPs. Maintenance of data investigation records that are reported to Corporate Management.
- Work with various organizations outside of TestAmerica to further the development of quality standards and represent TestAmerica at various trade meetings.
- Preparation of a monthly report that includes quality metrics across the Analytical Division and a summary of any quality related initiatives and issues.
- With the assistance of the Corporate Senior Management Teams and the EHS Directors, development and implementation of the TestAmerica Environmental, Health and Safety Program.

4.2.7 Quality Directors (Corporate)

The Quality Directors report to the VP-QA/EHS. Together with the VP-QA/EHS, the Quality Directors have the responsibility for the establishment, general overview and maintenance of

the Analytical Division's Quality Assurance Program within TestAmerica. The Quality Directors are responsible for:

- Oversight of the QA/QC programs within each laboratory. This includes a final review of each laboratory-specific QAM and receipt of each laboratory's QA monthly report.
- Review of QA/QC aspects of national projects.
- Assistance with certification activities.

4.2.8 Ethics and Compliance Officers (ECOs)

TestAmerica has designated two senior members of the Corporate staff to fulfill the role of Ethics and Compliance Officer (ECO) – VP-QA/EHS and VP-Client and Technical Services. Each ECO acts as a back-up to the other ECO and both are involved when data investigations occur. Each ECO has a direct line of communication to the entire senior Corporate and lab management staff.

The ECOs ensure that the organization distributes the data integrity and ethical practices policies to all employees and ensures annual trainings and orientation of new hires to the ethics program and its policies. The ECO is responsible for establishing a mechanism to foster employee reporting of incidents of illegal, unethical, or improper practices in a safe and confidential environment.

The ECOs monitor and audit procedures to determine compliance with policies and to make recommendations for policy enhancements to the CEOs, COOs, Laboratory Director/Manager or other appropriate individuals within the laboratory. The ECO will assist the laboratory QA Manager in the coordination of internal auditing of ethical policy related activities and processes within the laboratory, in conjunction with the laboratories regular internal auditing function.

The ECOs will also participate in investigations of alleged violations of policies and work with the appropriate internal departments to investigate misconduct, remedy the situation, and prevent recurrence of any such activity.

4.2.9 Vice President of Client and Technical Services

The Vice President (VP) of Client and Technical Services is responsible for offerings to clients including risk management, technical assistance, legal compliance and contract administration. The VP of Client and Technical Services provides support and direction to the Managers of these areas, and supports the COOs in decisions regarding long term planning, resource allocation and capital expenditures.

4.2.10 Director of Technical Services

The Director of Technical Services is responsible for establishing, implementing and communicating TestAmerica's Analytical Division's Technical Policies, SOPs, and Manuals. Other responsibilities include conducting technical assessments as required, acting as a technical resource in national contracts review, coordinating new technologies, establishing best practices, advising staff on technology advances, innovations, and applications.

4.2.11 Chief Information Officer (CIO)

The CIO is responsible for establishing, implementing and communicating TestAmerica Information Technology (IT) Policies, SOPs, and Manuals. Other responsibilities include coordinating new technologies; development of electronic communication tools such as TestAmerica intranet and internet sites; ensuring data security and documentation of software; ensuring compliance with the NELAC standard; and assistance in establishing, updating, and maintaining Laboratory Information Management Systems (LIMS) at the various TestAmerica facilities.

4.2.12 Environmental Health and Safety Directors (EHSDs) (Corporate)

The EHSDs report directly to the VP-QA/EHS. The EHSDs are responsible for the development and implementation of the TestAmerica Environmental, Health and Safety program. Responsibilities include:

- Consolidation and tracking all safety and health-related information and reports for the company, and managing compliance activities for TestAmerica locations.
- Coordination/preparation of the corporate Environmental, Health and Safety Manual Template that is used by each laboratory to prepare its own laboratory-specific Safety Manual/ CHP.
- Preparation of information and training materials for laboratory EHS Coordinators.
- Assistance in the internal and external coordination of employee exposure and medical monitoring programs to insure compliance with applicable safety and health regulations.
- Serving as Department of Transportation (D.O.T.) focal point and providing technical assistance to location management.
- Serving as Hazardous Waste Management main contact and providing technical assistance to location management.

4.2.13 Laboratory Director

- Reports directly to the Regional General Manager
- Responsible for implementation and adherence by lab staff to the TestAmerica North Canton QAM and all policies and procedures within the laboratory
- Has signature authority for QAM, policies, SOPs, and contracts (as detailed in TestAmerica policy)
- Assesses the effectiveness of the QAM within the operation
- Maintains adequate trained staff documented on organization charts
- Responsible for implementing internal/external audit findings corrective actions

4.2.14 Quality Assurance Manager

- Reports directly to the Laboratory Director and, for all QA matters, to the Corporate QA Director to maintain independence of QA oversight
- Responsible for the implementing and communicating the QAM
- Maintains, approves, and implements the QAM

- Has joint signature authority, with the Laboratory Director and Technical Director for approval of quality documents, e.g., QAM, policies, and SOPs
- Directs controlled distribution of laboratory quality documents
- Provides Quality System training to all new personnel
- Reviews and approves documentation of analyst training records
- Serves as a focal point for QA and QC issues, reviews corrective actions and recommends resolution for recurring nonconformances within the laboratory
- Assists in maintaining regulatory analytical compliance, including maintaining certifications, and in this regard has signature authority for laboratory quality documents
- Monitors data quality measures via statistical methods to verify that the laboratory routinely meets stated quality goals
- Performs systems, data, contract compliance, and surveillance audits.
- Hosts external audits conducted by outside agencies
- Responsible for approving quality control reference data changes in the LIMS
- Oversees the selection, review, and approval of analytical subcontractors
- Prepares monthly QA Reports to management describing significant quality events
- Has the final authority to accept or reject data and to stop work in progress in the event that procedures or practices compromise the validity and integrity of analytical data
- Responsible for implementing internal/external audit findings corrective actions

4.2.15 Operations Manager/Laboratory Supervisor

- Reports directly to the Laboratory Director
- Supervises daily activities of the Operational Groups
- Schedules analytical operations
- Supervises QC activities performed as a part of routine analytical operations
- Implements data review procedures
- Supervises the preparation and maintenance of laboratory records
- Supervises maintenance of instruments and scheduling of repairs
- Works with the Project Managers and Group Leaders to assure the requirements of projects are met in a timely manner
- Supervises daily activities of the Sample Control Group.

4.2.16 Laboratory Technical Director

- Reports directly to Laboratory Director
- Responsible for the technical operation of the laboratory
- Has joint signature authority for QAM, policies, SOPs, and training records
- Performs technical training in area(s) of expertise
- Interfaces with management on technical needs and solving day-to-day technical issues
- Investigates technical issues related to projects as directed by QA
- Evaluates new methods, technical proposals, and statements of work
- Certifies technical laboratory personnel based on education and background to ensure that staff have demonstrated capability in the activities for which they are responsible by reviewing and signing analyst demonstrations.
- Performs other tasks as required by NELAC.

The Technical Director meets the requirements specified in Section 4.1.1.1 of NELAC standards.

4.2.17 Project Management Group Leader

- Reports directly to the Manager of Client Services
- Supervises daily activities of the Project Management and Administrative Groups
- Works with the Operations Manager and/or Group/Team Leaders to ensure the requirements of projects are met in a timely manner

4.2.18 Manager of Client Services

- Reports directly to Laboratory Director
- Supervises Field Analytical Services Group
- Supervises Service Centers
- Supervises daily activities of the customer
- Supervises Service Managers

4.2.19 Customer Service Managers (CSMs)

- Reports directly to the Manager of Client Services
- Defines customer requirements through project definition
- Assesses and assures customer satisfaction
- Provides feedback to management on changing customer needs
- Brings together resources necessary to ensure customer satisfaction.

4.2.20 Project Manager

- Reports directly to the Project Manager Group Leader
- Monitors analytical and QA project requirements for a specified project
- Acts as a liaison between the client and the laboratory staff
- Communicates project-specific requirements to all parties involved
- Assists the laboratory staff with interpretation of work plans, contracts, and QAPP requirements
- Oversees project data packages for completeness and compliance to client needs
- Has signature authority for final reports
- Keeps the laboratory and client informed of project status
- Together with the QA Manager, approves customer requested variances to methods and to standard laboratory protocols
- Monitors, reviews, and evaluates the progress and performance of projects
- Reports client inquiries involving data quality issues or data acceptability to the facility QA Manager and to the operations staff
- Conducts project reviews to assess the laboratory's performance in meeting customer requirements
- Prepares reissue requests for project data
- Responsible for meeting quality requirements.

4.2.21 Group Leader

- Reports directly to the Operations Manager
- Supervises daily activities of analyses within the group
- Supervises QC activities performed as a part of routine analytical operations
- Implements data review procedures
- Supervises the preparation and maintenance of laboratory records
- Evaluates instrument performance and supervises the calibration, preventive maintenance, and scheduling of repairs
- Oversees or performs review and approval of all analytical data
- Reports nonconformances to the appropriate managers
- Responsible for generation of SOPs for their section
- Responsible for meeting quality requirements.

4.2.22 Analyst

- Performs analytical methods and data recording in accordance with documented procedures
- Performs and documents calibration and preventive maintenance
- Performs data processing and data review procedures
- Reports nonconformances to the Supervisor/Manager and QA Manager
- Ensures sample and data integrity by adhering to internal chain-of-custody procedures
- Responsible for meeting quality requirements defined in this LQM and other supporting QA procedures.

4.2.23 Sample Custodian

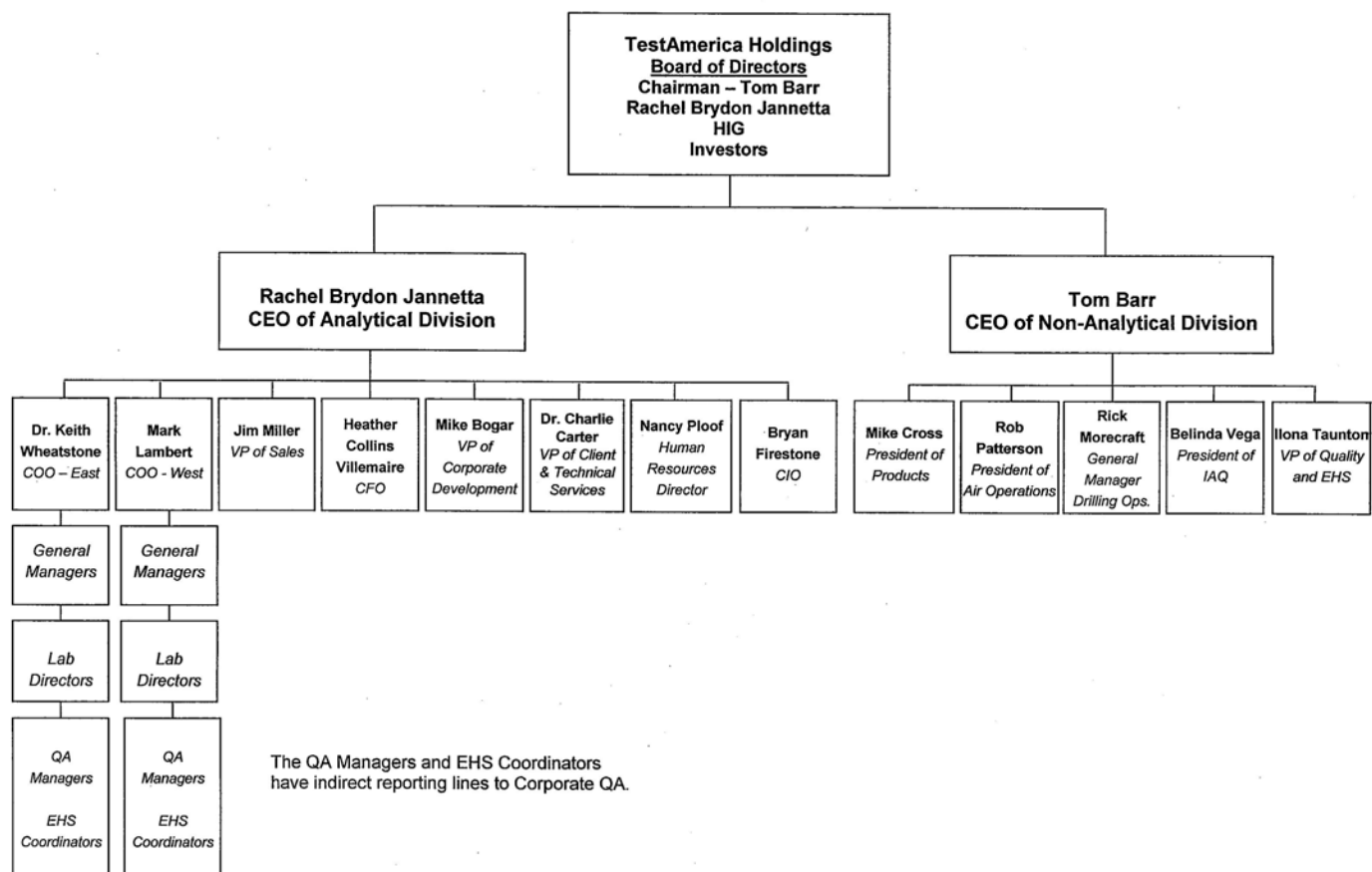
- Ensures implementation of proper sample receipt procedures, including maintenance of chain-of-custody
- Reports nonconformances associated with condition-upon-receipt of samples
- Logs samples into the LIMS
- Ensures that all samples are stored in the proper environment
- Assists Environmental Health and Safety staff with sample disposal
- Responsible for meeting quality requirements.

4.2.24 Report Production Staff

- Accurately generates and compiles analytical reports and associated deliverables for delivery to the client
- Responsible for meeting quality requirements
- Produce as needed reports that meet the NELAC requirements.

Figure 4-1.

Corporate Organization Chart



SECTION 5

QUALITY SYSTEM (NELAC 5.4.2)

5.1 QUALITY POLICY STATEMENT

The management of TestAmerica and TestAmerica North Canton are committed to providing data of known quality to its clients by adhering to approved methodologies, regulatory requirements and the QA/QC protocols described in this manual.

In all aspects of the laboratory and business operations, management is dedicated in maintaining the highest ethical standards. An Ethics Policy sign-off can be viewed in Appendix 1. Training on ethical and legal responsibilities is provided annually and each employee signs off annually on the policy as a condition of employment.

It is TestAmerica's Policy to continually improve systems and provide support to quality improvement efforts in laboratory, administrative and managerial activities. The company recognizes that the implementation of a quality assurance program requires management's commitment and support as well as the involvement of the entire staff.

TestAmerica North Canton strives to provide clients with the highest level of professionalism and the best service practices in the industry.

Every staff member at TestAmerica North Canton 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 seven elements of the TestAmerica Ethics and Data Integrity Program include:

- An Ethics Policy (Policy CA-L-P-001) and Employee Ethics Statements (Appendix 1)
- An Ethics and Compliance Officer (ECO)
- 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 (SOP CA-L-S-001)
- Procedures and guidance for recalling data if necessary (SOP CA-L-S-001)
- An effective external and internal monitoring system that includes procedures for internal audits (Section 16)

As an American Council of Independent Laboratories (ACIL) member, all TestAmerica laboratories adhere to the following ACIL Code of Ethics:

- 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 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 Quality System is communicated through a variety of documents prepared by the laboratory and company management:

- Quality Assurance Manual (QAM) Template
- 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 TestAmerica QA/QC Policy Memorandums (refer to Section 3.4).
- Laboratory QA/QC Policy Memorandums (refer to Section 3.4).

5.3.1 Order of Precedence

In the event of a conflict or discrepancy between policies, the order of precedence is as follows:

- TestAmerica QA/QC Policy Memorandum - Corporate
- Laboratory QA/QC Policy Memorandum
- Quality Assurance Manual
- Corporate SOPs and Policies

- Laboratory SOPs and Policies
- Other: Work Instructions (WI), memos, flow charts, etc.

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 will 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).

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, and by the degree to which approval from the US EPA or other pertinent regulatory agencies is obtained for any procedure for which significant modifications have been made.

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 will 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 prepares a Control Limit Report that summarizes the precision and accuracy acceptability limits for analyses performed at TestAmerica North Canton. This summary includes an effective date, is updated each time new limits are generated, and is located in QC Browser. The charts are stored as hard copies. 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 North Canton has developed limits from evaluation of data from similar matrices. Criteria for development of control limits is 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 [such as the Ohio Voluntary Action Plan (VAP)]. TestAmerica North Canton 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 will 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 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 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

The Corporate staff posts Corporate Manuals, SOPs, Policies, Work Instructions, White Papers and Training Materials on the company intranet site. These are collectively termed "Official Documents" and encompass the Policies and Procedures that all facilities are required to employ. These official 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 official documents is found in Corporate SOP CW-Q-S-001, Corporate Document Control and Archiving.

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 name 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 the system, and maintains the items electronically on the Laboratory public drive or the QA Department files.

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 as needed to those using it. Controlled documents shall be available at all locations where the operational activity described in the document is performed (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 will be reviewed at a minimum of every 24 months, and revised as appropriate. For procedures associated with DoD project work, SOPs and Policies are reviewed every 12 months. 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 SOPs NC-QA-002 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. Electronic copies are stored on the Public server in the QA folder for the applicable revision.

For changes to SOPs, refer to 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 as described in Section 15.

SECTION 7

REVIEW OF WORK REQUEST

7.1 OVERVIEW

TestAmerica North Canton 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 (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 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 will 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 review process 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 will 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 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 Managers
- 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.
- 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.

The contract will be distributed to and maintained by the Corporate Contracts Department and the applicable Account Executive. A copy of the contract will be filed electronically by the Laboratory Director. Quotes will 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, TestAmerica 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 direct 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).

TestAmerica 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.

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 corporate network. The phrase “work sharing” refers to internal transfers of samples between company 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 we must 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 will 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 will be placed with an appropriately accredited laboratory. The laboratory performing the subcontracted work will be identified in the final report, as will 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 will advise the client of a subcontract or work sharing arrangement in writing and when possible approval from the client shall 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 network 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 the company (in J.D.Edwards). 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 intra-company 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 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. 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 The QA Manager must ensure that the Subcontracting Approval Form (Figure 8-2) has been completed and have supporting documentation on file prior to initiation of any work. A letter or e-mail is sent to the lab requesting the following information:

8.2.1.1 If a lab is NELAC or A2LA-accredited:

8.2.1.1.1 Copy of necessary certifications verifying the required approvals are current. Ensure all needed analytes are included; some may not be accreditable (if so, document). Certificate and scope of international Standard accreditation are required, when applicable.

8.2.1.1.2 Insurance Certificate. This is required by the TestAmerica Chief Financial Officer

8.2.1.1.3 USDA soil permit, if available

8.2.1.2 For laboratories accredited by other agencies with an auditing program

8.2.1.2.1 Copy of necessary certifications verifying that the required approvals are current. Ensure all needed analytes are included; some may not be accreditable (if so, document). Certificate and scope of International Standard accreditation are required, when applicable.

8.2.1.2.2 Insurance Certificate. This is required by the TestAmerica Chief Financial Officer

8.2.1.2.3 USDA soil permit, if available**

8.2.1.2.4 Description of Ethics and Data Integrity Plan

8.2.1.2.5 The most recent two sets of full proficiency testing (PT) results relevant to the analyses of interest and any associated corrective action

8.2.1.2.6 State Audit with Corrective Action Response

8.2.1.2.7 Example final report to confirm format is compliant and provides the necessary information. (Minimally, it must be determined the Batch QC results are included in the laboratory reports, and data is appropriately qualified.)

8.2.1.2.8 A copy of raw data associated with the first project is requested for internal review. The raw data is reviewed by the QA Manager and the PM to ensure that the results meet the client's needs. If the QA Manager is unfamiliar with the analysis being performed, notify Corporate QA for guidance on the review. (It may need to be sent elsewhere for evaluation.) This requirement can be skipped if an on-site visit of the laboratory is planned. (This requirement is effective as of the effective date of this section. Laboratories worked with previously—minimum of six months—are

grandfathered.)

- 8.2.1.2.9** DoD work includes additional requirements as described in Section 8.1 above.
- 8.2.1.3** For laboratories performing tests that are unaccredited or accredited by an agency without an audit program:
 - 8.2.1.3.1** A copy of their Quality Assurance Manual (controlled, if possible). Ensure data quality limits for relevant methods are acceptable and that training procedures are adequate.
 - 8.2.1.3.2** A copy of necessary certifications (if available) verifying the required approvals are current. Ensure all needed analytes are included. Some may not be accreditable (if so, document). Certificate and scope of international Standard accreditation are required, when applicable.
 - 8.2.1.3.3** Insurance Certificate. This is required by the Test America Chief Financial Officer.
 - 8.2.1.3.4** USDA soil permit, if available**.
 - 8.2.1.3.5** Evidence of a current SOP per method. A copy of the first page and signature page of the SOP is acceptable. A Table of Contents including effective dates may also be acceptable. The SOP can be examined if an on-site audit is performed.
 - 8.2.1.3.6** Description of Ethics and Data Integrity Plan
 - 8.2.1.3.7** The most recent two sets of full proficiency testing (PT) results relevant to the analyses of interest and any associated corrective action.
 - 8.2.1.3.8** Example final report to confirm format is compliant and provides the necessary information. (Minimally, it must be determined the Batch QC results are included in the laboratory reports, and data is appropriately qualified.)
 - 8.2.1.3.9** Statement of Qualification (SOQ) or summary list of Technical Staff and Qualifications – position, education, and years of experience.
 - 8.2.1.3.10** DoD work includes additional requirements as described in Section 8.1 above.

**USDA permit is required if soils less than three feet deep from New York, North Carolina, South Carolina, Georgia, Florida, Tennessee, Alabama, Mississippi, Louisiana, Arkansas, Texas, Oklahoma, New Mexico, Arizona, California, Hawaii, or outside the continental U.S. are to be analyzed. These samples require special shipping measures (check with the EHS Department). It may be necessary to heat-treat the samples before shipping if the subcontract laboratory does not have a USDA permit; however, some analytes/tests may be irrelevant after heat treatment.

8.2.1.3.11 A copy of raw data associated with the first project is requested for internal review. The raw data is reviewed by the QA Manager and the PM to ensure that the results meet the client's needs. If the QA Manager is unfamiliar with the analysis being performed, notify Corporate QA for guidance on the review. (It may need to be sent elsewhere for evaluation.) This requirement can be skipped if an on-site visit of the laboratory is planned. (This requirement is effective as of the effective date of this section. Laboratories worked with previously—minimum of six months—are grandfathered.)

8.2.2 Once the information is received by the QA Manager, it is evaluated for acceptability and forwarded to Corporate Contracts for formal contracting with the laboratory. They will 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.

8.2.3 The client will 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. The company does not certify laboratories. The subcontractor is on our approved list, and can only be recommended to the extent that we would use them.

8.2.4 The status and performance of qualified subcontractors will be monitored periodically by the Corporate Contract Department. Any problems identified will be brought to Corporate QA attention.

- Complaints shall be investigated. Documentation of the complaint, investigation, and corrective action will be maintained in the subcontractor file on the intranet site. Complaints must be posted using the Vendor Performance Report (Form CW-F-WI-009).
- Information must be updated on the intranet when new information is received from the subcontracted laboratories.
- Subcontractors in good standing will be retained on the intranet listing. The QA Manager will notify all network laboratories and Corporate QA and Corporate Contracts if any laboratory requires removal from the intranet site. This notification will be posted on the intranet site and e-mailed to all Lab Directors/Managers, QA Managers, and Sales Directors.

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. The information is documented on a Subcontracted Sample Form (Figure 8-3) and the form is retained in the project folder. For network laboratories, certifications can be viewed on the company website.

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 network.

The PM will communicate with the subcontracted laboratory to monitor the status of the analyses, facilitate successful execution of the work and ensure 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 network work sharing laboratory may be transferred electronically and the results reported by the network 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, Corporate QA must be informed, and the QA Manager will be required to verify adequacy of proficiency scores and certifications. The laboratory must also request a copy of the raw data to support the analytical results for the first project submitted to the subcontract laboratory unless the laboratory has NELAC accreditation. The raw data is reviewed by the QA Manager and the PM to ensure that the results meet the client's needs. The QA Manager will request full documentation and qualify the subcontractor under the provisions above. The approval process should be completed 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

Figure 8-2.

Example - Subcontracting Laboratory Approval Form (Initial / Renewal)

SUBCONTRACTING LABORATORY APPROVAL

Reference: Section 8 – Quality Assurance Manual

Date: _____
Laboratory: _____
Address: _____
Contact and e-mail address: _____
Phone: Direct _____ Fax _____

Requested Item ³	Date Received	Reviewed/ Accepted	Date
1. QA Manual ³			
2. Copy of State Certification ¹			
3. State Audit with Corrective Action Response (or NELAC or A2LA Audit) ³			
4. Most Recent (and relevant) 2 Sets of WP/WS Reports with Corrective Action Response ^{1,3}			
5. SOQ or Summary list of Technical Staff and Qualifications ³			
6. SOPs for Methods to Be Loadshifted ^{2,3}			
7. USDA Soil Permit			
8. Insurance Certificate			
9. Sample Report ³			
10. For DoD Work: Statement that Lab quality system complies with QSM			
11. For DoD Work: Approved by specific DoD Component laboratory approval process			
11. Description of Ethics Program ³			

1 - Required when emergency procedures are implemented.

2 - Some labs may not submit copies due to internal policies. In these cases, a copy of the first page and signature page of the SOP is acceptable. This requirement may also be fulfilled by supplying a table of SOPs with effective dates.

3 – If the laboratory has NELAC accreditation, Item numbers 4 through 10 are not required.

On Site Audit Planned: YES NO If yes, Date Completed: _____ By Whom: _____

Comments: _____

Lab Acceptable for Subcontracting Work: YES NO Limitations: _____

QA Manager: _____ Date: _____
(Printed Name)

☐ Forwarded to Contract Coordinator by: _____ 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, all purchases from specific vendors are approved by a member of the supervisory or management staff.

Capital expenditures are made in accordance with the Controlled Purchases Procedure, CW-F-S-004. Only one quote is required where the item being purchased is a sole source product. Examples of sole source capital expenditures are laboratory test equipment, client specified purchases and building leases. A minimum of two quotes is required where the opportunity exists to source from more than one vendor. All documentation related to the purchase of capital items will be maintained in the individual CapEx files located in Corporate Purchasing. Data will be held in accordance with the record retention policy.

TestAmerica will enter into formal contracts with vendors when it is advantageous to do so. Contracts will be signed in accordance with the Authorization Matrix Policy, CW-F-P-002. Examples of items that are purchased through vendor contracts are laboratory instruments, consumables, copiers and office supplies. Request for Proposals (RFP's) will be issued where more information is required from the potential vendors than just price. 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 according to laboratory procedure. Pyrex (or equivalent) glass should be used where possible. For safety purposes, thick-wall glassware should be used where available.

9.3 REAGENTS, STANDARDS & SUPPLIES

Chemical reagents, solvents, glassware, and general supplies are ordered as needed to maintain sufficient quantities on hand. Purchasing guidelines for equipment and reagents must

meet with 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 CA-Q-S-001.

9.3.1 Purchasing

The nature of the analytical laboratory demands that all material used in any of the procedures is 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.

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 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

There are many different grades of analytical reagents available to the analyst. 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. If 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.

- 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 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 at 25°C. The conductivity is checked and recorded daily. If the water's conductivity is less than the specified limit, the Operations Manager 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) 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. Table 9-1 details specific 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 supervisor makes a supply request to the Operations Manager and/or the Laboratory Director/Manager. If they agree with the request the procedures outlined in 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, it is given a short name, 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 it can be linked 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 (see 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 as specified in the laboratory's procedure for software verification. 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 21. 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). The level of control used in the selection process is dependent on the anticipated spend 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 will work through the appropriate channels to gather the information required to clearly identify the problem and will 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 will 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 (CW-F-WI-007 – refer to Figure 9-2).

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.

Table 9-1.
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 should not be stored with bases.
Bulk Dry Chemicals	Stored in the original containers at room temperature. All organic acids must be stored separately from inorganic acids. Acids should not be stored with bases.
Working Solutions containing Organic Compounds	Stored as per method recommendation/ requirement. They are generally stored refrigerated at 4°C± 2°C.
Working Solutions containing only Inorganics	Stored at room temperature; refrigeration is optional.
Flammable Solvents	Stored in solvent cabinets at room temperature.
Non-Flammable Solvents	Stored separately from the flammable solvents in cabinets at room temperature.

Figure 9-2.
Example – JD Edwards Vendor Add Request Form



JD Edwards Vendor Add Request Form

Vendor name:	Lab location <u>and</u> 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 (<u>Required for Aerotech / P&K only</u>)	

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 North Canton 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 (Sections 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. When requested the following special services are provided:

- The laboratory will provide the client or the client's representative reasonable access to the relevant areas of the laboratory for the witnessing of tests performed for the client.
- The laboratory will work with client-specified third party data validators as specified in the client's contract.
- The laboratory will provide the client with all requested information pertaining to the analysis of their samples. 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

Customer Service Managers (CSMs) and Project Managers (PMs) are an important communication link to the clients. The lab shall inform its clients of any delays in project completion as well as any non-conformances in either sample receipt (refer to Section 24) or sample analysis. Project Management will maintain ongoing client communication throughout the entire client project.

A Technical Director, Operation Manager, or Lab Supervisors are available to discuss any technical questions or concerns that the client may have.

10.4 REPORTING

The laboratory will work 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 Sales and Marketing teams periodically develop lab and client-specific surveys to assess client satisfaction.

SECTION 11

COMPLAINTS (NELAC 5.4.8)

11.1 OVERVIEW

TestAmerica North Canton believes that effective client complaint handling processes have important business and strategic value. Listening to and documenting client concerns captures 'client knowledge' that helps 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, 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 dealing with both external and internal complaints.

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 following SOPs CORP-QA-0010, Nonconformance and Corrective Action System, and S-C-002, Complaint Handling and Service Recovery. It is the laboratory's goal to provide a satisfactory resolution to complaints in a timely and professional manner.

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 should 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 Complaints
- Complaint Investigation and Service Recovery

- Process Improvement

The laboratory shall inform 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 and shall follow the procedures outlined in Section 13. 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 13.

11.4 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 17)

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. Refer to SOP CORP-QA-0010, Nonconformance and Corrective Action System, for details of the nonconformance process.

12.2 RESPONSIBILITIES AND AUTHORITIES

SOP CA-L-S-001, Internal Investigation of Potential Data Discrepancies and Determination for Data Recall, outlines the general procedures for the reporting and investigation of data discrepancies and alleged incidents of misconduct or violations of the company'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 will 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 laboratory management within 24 hours. The Senior Management staff is comprised 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) and Quality Director within 24 hours.

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, COOs (East and West), General Managers and the Quality Directors (East and West) 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.

SOP CA-L-S-001 distinguishes between situations when it would be appropriate for the laboratory QA Manager and Laboratory Director/Manager (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 SOP 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 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.

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 will be respected, and the problem and the required corrective and preventive action will be stated in writing and presented to the Laboratory Director/Manager.

The Laboratory Director/Manager shall arrange for the appropriate personnel to meet with the QA Manager as needed. This meeting shall be held to confirm that there is a problem, that suspension/restriction of the method is required and will 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 will also initiate 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 must 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 will 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 will 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, Supervisor) can devise a start-up plan to cover all steps from client notification through compliance and release of reports. Project Management, Director of Client Services, and Director of Sales and Marketing should 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 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 Nonconformance Memos (NCM).

13.2 DEFINITIONS

- **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 are contained in the method-specific SOPs. 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 is not only a correction made to the immediate event, but a change in process, procedure or behavior that is required to eliminate the causes of an existing nonconformity, defect, or other undesirable situation in order to prevent recurrence.

13.3 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).

13.3.1 Non-Conformance Memo (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

13.4 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.

13.4.1 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 13-1 in SOP CORP-QA-0010, Nonconformance and Corrective Action System, provides some general guidelines on determining responsibility for assessment.
- 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 Supervisor, Lab Director, QA Manager, or designee is consulted.

13.4.2 Selection and Implementation of Corrective Actions

- Where corrective action is needed, the laboratory shall 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 shall 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 shall document and implement the changes. The NCM is used for this documentation.

13.4.3 Monitoring of the Corrective Actions

- The Department Manager/Supervisor and QA Manager is responsible to ensure that the corrective action taken was effective.
- Ineffective actions will be 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 that 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 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 QA Manager, indicating the nature of the out-of-control situation and problems encountered in solving the situation.

13.4.4 Follow-up Audits

- Follow-up audits may be initiated by the QA Manager and shall 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. (Section 16 includes additional information regarding internal audit procedures.)
- 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.

13.5 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 NCM.

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 should 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 at a minimum monthly by the QA Manager and highlights are included in the QA monthly report.

To the extent possible, samples shall be reported only if all quality control measures are acceptable. If the deficiency does not impair the usability of the results, data will be reported with an appropriate data qualifier and/or the deficiency will 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., reanalysis) is taken and documented.

13.6 BASIC CORRECTIONS

When mistakes occur in records, each mistake shall 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 shall 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) shall also be documented.

Table 13-1.
Example – General Corrective Action Procedures

* For Ohio VAP, method blank contamination must not exceed the RL if that analyte is a Contaminant of Concern for the project site.

INORGANIC LABORATORY QUALITY CONTROL SAMPLES

Analysis	QC Sample	Method	NPDES ¹	Method	RCRA (SW846) ²
Alkalinity	* Method Blank	310.1 2320B	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Concentration must be less than the reporting limit <u>Corrective Action:</u> Rerun all samples associated with unacceptable blank	—	N/A
	Laboratory Control Sample	310.1 2320B	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Percent recovery must be within laboratory control limits <u>Corrective Action:</u> If not within laboratory control limits, rerun all associated samples	—	N/A
	Matrix Spike	310.1 2320B	<u>Total alkalinity:</u> 1 per batch of 20 samples	—	N/A
	Matrix Spike Duplicate	310.1 2320B	<u>Total alkalinity:</u> 1 per batch of 20 samples	—	N/A
	Duplicate	310.1 2320B	For carbonate, bicarbonate, hydroxide, alkalinity only. <u>Frequency:</u> 1 per batch of 10 samples <u>Criteria 310.1:</u> ≤ 20 % RPD ⁽³⁾ <u>Criteria 2320B:</u> ≤ 25 % RPD ⁽³⁾ <u>Corrective Action:</u> 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	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Concentration less than reporting limit <u>Corrective Action:</u> Rerun all samples associated with unacceptable blank	—	N/A
	Laboratory Control Sample	350.1 350.2 SM4500 NH-E,F	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Percent recovery must be within laboratory control limits <u>Corrective Action:</u> If not within control limits, rerun all associated samples	—	N/A
	Matrix Spike	350.1 350.2 SM4500 NH-E,F	<u>Frequency:</u> 1 per 20 samples, minimum of one per batch of samples processed <u>Criteria:</u> Percent recovery must be within laboratory control limits <u>Corrective Action:</u> 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	<u>Frequency:</u> 1 per 20 samples, minimum of one per batch of samples processed <u>Criteria:</u> Percent recovery must be within laboratory control limits <u>Corrective Action:</u> 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	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Concentration must be less than the reporting limit <u>Corrective Action:</u> 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	NPDES¹	Method	RCRA (SW846)²
BOD	* Method Blank	405.1 SM5210B	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Concentration must be less than the reporting limit <u>Corrective Action:</u> Rerun all samples associated with unacceptable blank	—	N/A
	Laboratory Control Sample and Laboratory Control Sample Duplicate	405.1 SM5210B	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Percent recovery must be within laboratory control limits <u>Corrective Action:</u> 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	NPDES¹	Method	RCRA (SW846)²
Bromide	* Method Blank	300.0A ⁽⁵⁾	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Concentration must be less than the reporting limit <u>Corrective Action:</u> Rerun all samples associated with unacceptable blank	9056A	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Concentration must be less than the reporting limit <u>Corrective Action:</u> Rerun all samples associated with unacceptable blank
	Laboratory Control Sample and Laboratory Control Sample Duplicate	300.0A ⁽⁵⁾	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Percent recovery must be within laboratory control limits <u>Corrective Action:</u> If not within control limits, rerun all associated samples	9056A	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Percent recovery must be within laboratory control limits <u>Corrective Action:</u> If not within control limits, rerun all associated samples
	Matrix Spike	300.0A ⁽⁵⁾	<u>Frequency:</u> 1 per 10 samples, minimum of one per batch of samples processed <u>Criteria:</u> Percent recovery must be within laboratory control limits <u>Corrective Action:</u> Flag data outside of limit	9056A	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Percent recovery must be within laboratory control limits <u>Corrective Action:</u> 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	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Concentration must be less than the reporting limit <u>Corrective Action:</u> Rerun all samples associated with unacceptable blank	—	N/A
	Laboratory Control Sample and Laboratory Control Sample Duplicate	410.4 SM5220D	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Percent recovery must be within laboratory control limits <u>Corrective Action:</u> If not within laboratory control limits, rerun all associated samples	—	N/A
	Matrix Spike	410.4 SM5220D	<u>Frequency:</u> 1 per 10 samples, minimum of one per batch of samples processed <u>Criteria:</u> Must be within laboratory control limits <u>Corrective Action:</u> Flag data outside of limit	—	N/A
	Matrix Spike Duplicate	410.4 SM5220D	<u>Frequency:</u> 1 per 20 samples, minimum of one per batch of samples processed <u>Criteria:</u> Must be within laboratory control limits <u>Corrective Action:</u> 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	<u>Frequency</u> : 1 with each batch of samples processed not to exceed 20 samples <u>Criteria</u> : Concentration must be less than the reporting limit <u>Corrective Action</u> : Rerun all samples associated with unacceptable blank	9056A	<u>Frequency</u> : 1 with each batch of samples processed not to exceed 20 samples <u>Criteria</u> : Concentration must be less than the reporting limit <u>Corrective Action</u> : Rerun all samples associated with unacceptable blank
	Laboratory Control Sample	300.0A ⁵ 325.2 325.3	<u>Frequency</u> : 1 with each batch of samples processed not to exceed 20 samples <u>Criteria</u> : Percent recovery must be within laboratory control limits <u>Corrective Action</u> : If not within control limits, rerun all associated samples	9056A	<u>Frequency</u> : 1 with each batch of samples processed not to exceed 20 samples <u>Criteria</u> : Percent recovery must be within laboratory control limits <u>Corrective Action</u> : If not within laboratory control limits, rerun all associated samples
	Matrix Spike	300.0A ^(b)	<u>Frequency</u> : 1 per 10 samples, minimum of one per batch of samples processed <u>Criteria</u> : Percent recovery must be within laboratory control limits <u>Corrective Action</u> : Flag data outside of limit	9056A	<u>Frequency</u> : 1 with each batch of samples processed not to exceed 20 samples <u>Criteria</u> : Percent recovery must be within laboratory control limits <u>Methods 9251 Corrective Action</u> : If not within laboratory control limits, rerun all associated samples <u>Method 9056/9253</u> <u>Corrective Action</u> : 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	<u>Frequency:</u> 1 per 20 samples, minimum of one per batch of samples processed <u>Criteria:</u> Percent recovery must be within laboratory control limits <u>Corrective Action:</u> Flag data outside of limit	9056A	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Percent recovery must be within laboratory control limits/< 20 % RPD ⁽³⁾ <u>Corrective Action:</u> Flag data outside of limit <u>Method 9056:</u> MSD is not applicable
	Duplicate	300.0A ⁽⁵⁾ 325.2 325.3	N/A	9056A	N/A
Chlorine, Residual	* Method Blank	330.5	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Concentration must be less than the reporting limit <u>Corrective Action:</u> 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	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> ≤ 20 % RPD ⁽³⁾ <u>Corrective Action:</u> 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	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Concentration must be less than the reporting limit <u>Corrective Action:</u> Rerun all samples associated with unacceptable blank	7196A 3060A	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Concentration less than reporting limit <u>Corrective Action:</u> Rerun all samples associated with unacceptable blank
	Laboratory Control Sample	3500 Cr-D	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Percent recovery must be within laboratory control limits <u>Corrective Action:</u> If not within laboratory control limits, rerun all associated samples	7196A 3060A	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples prepped <u>Criteria:</u> percent recovery for water must be within ± 15 % and for solids must be within ± 20% <u>Corrective Action:</u> Rerun all samples associated with unacceptable LCS
	Matrix Spike	3500 Cr-D	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Must be within laboratory QC limits <u>Corrective Action:</u> Flag data outside of limit	3060A 7196A	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Advisory limits are 75% - 125% recovery <u>Corrective Action:</u> Flag data associated with unacceptable Matrix Spike

INORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES¹	Method	RCRA (SW846)²
Chromium (Cr ⁺⁶) (Cont'd)	Matrix Spike Duplicate	3500 Cr-D	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Must be within laboratory QC limits <u>Corrective Action:</u> Flag data outside of limit	7196A 3060A	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Advisory limits are 75% - 125% recovery <u>Corrective Action:</u> Flag data associated with unacceptable Matrix Spike
	Duplicate	3500 Cr-D	N/A	7196A 3060A	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> ≤ 20 % RPD ⁽³⁾ limit <u>Corrective Action:</u> 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: $\leq 20\%$ 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	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Concentration must be less than the reporting limit <u>Corrective Action:</u> Rerun all samples associated with unacceptable blank	9012A	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Concentration less than reporting limit <u>Corrective Action:</u> Rerun all samples associated with unacceptable blank
	Laboratory Control Sample	335.1 SM4500C N-G	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Percent recovery must be within laboratory control limits <u>Corrective Action:</u> If not within laboratory control limits, rerun all associated samples	9012A	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Percent recovery must be within laboratory control limits <u>Corrective Action:</u> 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	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Percent recovery must be within lab control limits <u>Corrective Action:</u> 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	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Concentration must be less than the reporting limit <u>Corrective Action:</u> Rerun all samples associated with unacceptable blank	9012A	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Concentration less than reporting limit <u>Corrective Action:</u> Rerun all samples associated with unacceptable blank
	Laboratory Control Sample	335.2 335.3 335.4 4500-CN E	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Percent recovery must be within laboratory control limits <u>Corrective Action:</u> If not within laboratory control limits, rerun all associated samples	9012A	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Percent recovery must be within laboratory control limits <u>Corrective Action:</u> Rerun all samples associated with unacceptable LCS
	Matrix Spike	335.2 335.3 335.4 4500-CN E	<u>Frequency:</u> 1 per 20 samples, minimum of one per batch of samples processed <u>Criteria:</u> Percent recovery must be within laboratory control limits <u>Corrective Action:</u> Flag data outside of limit	9012A	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Percent recovery must be within laboratory control limits <u>Corrective Action:</u> 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	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	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	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Concentration must be less than the reporting limit <u>Corrective Action:</u> Rerun all samples associated with unacceptable blank	9056A	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Concentration must be less than the reporting limit <u>Corrective Action:</u> Rerun all samples associated with unacceptable
	Laboratory Control Sample	300.0A ⁽⁵⁾ 340.2 SM4500F-C,ISE	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Percent recovery must be within laboratory control limits <u>Corrective Action:</u> If not within laboratory control limits, rerun all associated samples	9056A	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Percent recovery must be within laboratory control limits <u>Corrective Action:</u> If not within control limits, rerun all associated samples
	Laboratory Control Sample Duplicate	300.0A ⁽⁵⁾ SM4500F-C,ISE	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Percent recovery must be within laboratory control limits <u>Corrective Action:</u> If not within laboratory control limits, rerun all associated samples	9056	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Percent recovery must be within laboratory control limits <u>Corrective Action:</u> 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	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Concentration must be less than the reporting limit <u>Corrective Action:</u> Rerun all samples associated with unacceptable blank	—	N/A
	Laboratory Control Sample	130.2 2340B	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Percent recovery must be within laboratory control limits <u>Corrective Action:</u> If not within laboratory control limits, rerun all associated samples	—	N/A
	Matrix Spike	130.2 2340B	<u>Method 130.2:</u> 1 per 20 samples <u>Method 2340B:</u> <u>Frequency , Criteria, and Corrective Action:</u> 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	<u>Method 130.2:</u> 1 per 20 samples <u>Method 2340B:</u> <u>Frequency, Criteria, and Corrective Action:</u> 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	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Concentration must be less than the reporting limit <u>Corrective Action:</u> Rerun all samples associated with unacceptable blank	—	N/A
	Laboratory Control Sample	3500-Fe D	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Percent recovery must be within laboratory control limits <u>Corrective Action:</u> 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	<u>Frequency</u> : 1 every 10 samples <u>Criteria</u> : Must be within laboratory QC limits <u>Corrective Action</u> : Flag associated data outside of limit	—	N/A
	Matrix Spike Duplicate	3500-Fe D	<u>Frequency</u> : 1 every 10 samples <u>Criteria</u> : Must be within laboratory QC limits <u>Corrective Action</u> : 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	<u>Frequency</u> : 1 with each batch of samples processed not to exceed 20 samples <u>Criteria</u> : Concentration must be less than the reporting limit <u>Corrective Action</u> : Rerun all samples associated with unacceptable blank	9056A	<u>Frequency</u> : 1 with each batch of samples processed not to exceed 20 samples <u>Criteria</u> : 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 <u>Criteria:</u> Percent recovery must be within laboratory control limits <u>Corrective Action:</u> If not within laboratory control limits, rerun all associated samples	9056A	Frequency: 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Percent recovery must be within laboratory control limits <u>Corrective Action:</u> 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 <u>Criteria:</u> Must be within laboratory control limits <u>Corrective Action:</u> Flag data outside of limit	9056A	Frequency: 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Percent recovery must be within laboratory control limits <u>Corrective Action:</u> 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 <u>Criteria:</u> Must be within laboratory control limits <u>Corrective Action:</u> 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	<u>Frequency:</u> 1 per 10 samples <u>Criteria:</u> RPD ⁽³⁾ must be within laboratory control limits <u>Corrective Action:</u> If not within laboratory control limits, flag all associated samples
Nitrite	* Method Blank	300.0A ⁽⁵⁾ 353.2	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Concentration must be less than the reporting limit <u>Corrective Action:</u> Rerun all samples associated with unacceptable blank	9056A	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Concentration must be less than the reporting limit <u>Corrective Action:</u> Rerun all samples associated with unacceptable blank
	Laboratory Control Sample	300.0A ⁽⁵⁾ 354.1 353.2	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Percent recovery must be within laboratory control limits <u>Corrective Action:</u> If not within laboratory control limits, rerun all associated samples	9056A	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Percent recovery must be within laboratory control limits <u>Corrective Action:</u> 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	<u>Frequency:</u> 1 per 10 samples, minimum of one per batch of samples processed <u>Criteria:</u> Must be within laboratory control limits <u>Corrective Action:</u> Flag data outside of limit	9056A	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Percent recovery must be within laboratory control limits <u>Corrective Action:</u> If not within laboratory control limits, flag all associated samples
	Matrix Spike Duplicate	300.0A ⁽⁵⁾ 354.1 353.2	<u>Frequency:</u> 1 per 20 samples, minimum of one per batch of samples processed <u>Criteria:</u> Must be within laboratory control limits <u>Corrective Action:</u> Flag data outside of limit	9056A	N/A
	Duplicate	300.0A ⁽⁵⁾ 354.1 353.2	N/A	9056A	<u>Frequency:</u> 1 per 10 samples <u>Criteria:</u> RPD ⁽³⁾ must be within laboratory control limits <u>Corrective Action:</u> 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: $\leq 20\%$ 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 $\leq 20\%$ 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	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Percent recovery must be within laboratory control limits <u>Corrective Action:</u> If not within laboratory control limits, rerun all associated samples	9065	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Percent recovery must be within laboratory control limits <u>Corrective Action:</u> If not within laboratory control limits, rerun all associated samples
	Matrix Spike	420.1	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Percent recovery must be within laboratory control limits <u>Corrective Action:</u> Flag data associated with unacceptable matrix spike	9065	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Percent recovery must be within laboratory control limits <u>Corrective Action:</u> Flag associated data
	Matrix Spike Duplicate	420.1	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Percent recovery must be within laboratory control limits <u>Corrective Action:</u> Flag associated data	9065	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Percent recovery must be within laboratory control limits <u>Corrective Action:</u> 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	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Concentration must be less than the reporting limit <u>Corrective Action:</u> Rerun all samples associated with unacceptable blank	9056A	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Concentration less than reporting limit <u>Corrective Action:</u> Rerun all samples associated with unacceptable blank
	Laboratory Control Sample	300.0 ^(4,5) 365.3 365.1 SM4500P-E	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Percent recovery must be within laboratory control limits <u>Corrective Action:</u> If not within laboratory control limits, rerun all associated samples	9056A	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Percent recovery must be within laboratory control limits <u>Corrective Action:</u> If not within laboratory control limits, rerun all associated samples
	Matrix Spike	300.0 ^(4,5) 365.3 365.1 SM4500P-E	<u>Frequency:</u> 1 per 10 samples <u>Criteria:</u> Must be within laboratory QC limits <u>Corrective Action:</u> Flag data outside of limit	9056A	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Percent recovery must be within laboratory control limits <u>Corrective Action:</u> Flag associated data associated with MS outside of limits
	Matrix Spike Duplicate	365.3 365.1 SM4500P-E	<u>Frequency:</u> 1 per 20 samples <u>Criteria:</u> Must be within laboratory QC limits <u>Corrective Action:</u> 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	<u>Frequency</u> : 1 with each batch of samples processed <u>Criteria</u> : RPD ⁽³⁾ must be within laboratory control limits <u>Corrective Action</u> : 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	<u>Frequency</u> : 1 with each batch of samples processed not to exceed 20 samples <u>Criteria</u> : Concentration must be less than the reporting limit <u>Corrective Action</u> : If analyte level in method blank is $\geq$ 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	<u>Frequency</u> : 1 with each batch of samples processed not to exceed 20 samples <u>Criteria</u> : Percent recovery must be within laboratory control limits <u>Corrective Action</u> : 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	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 10 samples <u>Criteria:</u> Sample results should agree within 20% if both the sample and sample duplicate results are > 5 X RL <u>Corrective Action:</u> 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	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Concentration must be less than the reporting limit <u>Corrective Action:</u> Rerun all samples associated with unacceptable blank	9038 9056A	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Concentration must be less than the reporting limit <u>Corrective Action:</u> Rerun all samples associated with unacceptable blank
	Laboratory Control Sample	300.0A ⁽⁵⁾ 375.4	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Percent recovery must be within laboratory control limits <u>Corrective Action:</u> If not within laboratory control limits, rerun all associated samples	9038 9056A	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Method 9038 Criteria:</u> Percent recovery must be within $\pm 15\%$ <u>Method 9056 Criteria:</u> Percent recovery must be within laboratory control limits <u>Corrective Action:</u> 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	<u>Frequency:</u> 1 per 10 samples, minimum of one per batch of samples processed <u>Criteria:</u> Percent recovery must be within laboratory control limits <u>Corrective Action:</u> Flag data outside of limit	9038 9056A	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 10 samples (9038) or 20 samples (9056) <u>Method 9038 Criteria:</u> Limits are 75% - 125% recovery <u>Method 9056 Criteria:</u> Percent recovery must be within laboratory control limits <u>Corrective Action:</u> Flag data associated with unacceptable Matrix Spike
	Matrix Spike Duplicate	300.0A ⁽⁵⁾ 375.4	<u>Frequency:</u> 1 per 20 samples, minimum of one per batch of samples processed <u>Criteria:</u> Percent recovery must be within laboratory control limits <u>Corrective Action:</u> 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	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Concentration must be less than the reporting limit <u>Corrective Action:</u> Rerun all samples associated with unacceptable blank	9030A	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Concentration must be less than the reporting limit <u>Corrective Action:</u> 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	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Percent recovery must be within laboratory control limits <u>Corrective Action:</u> If not within laboratory control limits, rerun all associated samples	9030A	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Percent recovery must be within laboratory control limits <u>Corrective Action:</u> Flag associated data
	Matrix Spike	376.1	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Percent recovery must be within laboratory control limits <u>Corrective Action:</u> Flag data outside of limit	9030A	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Percent recovery must be within laboratory control limits <u>Corrective Action:</u> Flag associated data
	Matrix Spike Duplicate	376.1	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Percent recovery must be within laboratory control limits <u>Corrective Action:</u> Flag data outside of limit	9030A	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Percent recovery must be within laboratory control limits <u>Corrective Action:</u> 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	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Concentration must be less than the reporting limit <u>Corrective Action:</u> Rerun all samples associated with unacceptable blank	9060 Walkley-Black	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Concentration less than reporting limit <u>Corrective Action:</u> Rerun all samples associated with unacceptable blank
	Laboratory Control Sample	415.1 SM5310D	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Percent recovery must be within laboratory control limits <u>Corrective Action:</u> If not within laboratory control limits, rerun all associated samples	9060 Walkley-Black	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> percent recovery must be within laboratory control limits <u>Corrective Action:</u> Rerun all samples associated with unacceptable LCS
	Matrix Spike	415.1 SM5310D	<u>Frequency:</u> 1 per 20 samples, minimum of one per batch of samples processed <u>Criteria:</u> Percent recovery must be within laboratory control limits <u>Corrective Action:</u> Flag data outside of limit	9060 Walkley-Black	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Percent recovery must be within laboratory control limits <u>Corrective Action:</u> 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	<u>Frequency:</u> 1 per 20 samples, minimum of one per batch of samples processed <u>Criteria:</u> Percent recovery must be within laboratory control limits <u>Corrective Action:</u> Flag data outside of limit	9060 Walkley-Black	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Percent recovery must be within laboratory control limits <u>Corrective Action:</u> 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⁾	<u>Frequency:</u> 1 with each set of 8 samples <u>Criteria:</u> Concentration less than reporting limit <u>Corrective Action:</u> Rerun all samples associated with unacceptable blank	9020B	<u>Frequency:</u> Run in duplicate between each group of 8 analytical determinations <u>Criteria:</u> Concentration less than reporting limit or less than 2 X MDL or RL whichever is lower <u>Corrective Action:</u> Rerun all samples associated with unacceptable blank
	Laboratory Control Sample	450.1 ⁽⁵⁾	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Percent recovery of analyte must be within laboratory control limits <u>Corrective Action:</u> Rerun all samples associated with unacceptable LCS (ICV)	9020B	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Percent recovery of analyte must be within 90-110% <u>Corrective Action:</u> 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⁾	<u>Frequency:</u> 1 per 20 samples, minimum of one per batch of samples processed <u>Criteria:</u> Must be within laboratory control limits <u>Corrective Action:</u> Reanalyze if sample remaining. If not, flag data with unacceptable Matrix Spike	9020B	<u>Frequency:</u> 1 per batch of 10 samples <u>Criteria:</u> Must be within laboratory control limits <u>Corrective Action:</u> Flag data associated with unacceptable Matrix Spike SOP NO. CORP-WC-0001
	Matrix Spike Duplicate	450.1 ⁽⁵⁾	<u>Frequency:</u> 1 per 20 samples, minimum of one per batch of samples processed <u>Criteria:</u> Must be within laboratory control limits <u>Corrective Action:</u> 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	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Concentration must be less than the reporting limit <u>Corrective Action:</u> 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	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Percent recovery must be within laboratory control limits <u>Corrective Action:</u> 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	<u>Frequency:</u> 1 per 10 samples <u>Criteria:</u> Must be within laboratory QC limits <u>Corrective Action:</u> Flag data outside of limit Not Applicable.	—	N/A
Mercury by CVAA & CVAFS	* Method Blank	200 series 1631E	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Concentration less than reporting limit <u>Corrective Action:</u> Rerun all samples associated with unacceptable blank SOP CORP-MT-0003. Exception: If blank is above RL, and samples are ND.	7000A series	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Concentration less than reporting limit <u>Corrective Action:</u> Rerun all samples associated with unacceptable blank SOP CORP-MT-0003. Exception: If 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	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> percent recovery of analyte must be within ± 20 %. 1631E is $\pm 23\%$ <u>Corrective Action:</u> Rerun all samples associated with unacceptable LCS SOP CORP-MT-0003. Exception: If samples are ND, results are reported.	7000A series	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> percent recovery of analyte must be within ± 20 % <u>Corrective Action:</u> Rerun all samples associated with unacceptable LCS SOP CORP-MT-0003. Exception: If samples are ND, results are reported.
	Matrix Spike	200 series 1631E	<u>Frequency:</u> with each batch of samples processed not to exceed 20 samples. 1631E frequency is 1 in 10 samples, 71-125% <u>Criteria:</u> Recovery should be within 75-125 % <u>Corrective Action:</u> Flag data associated with unacceptable MS. (See SOP CORP-MT-0003 for detailed corrective action procedure and for other QC procedures.)	7000A series	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Recovery should be within 75-125 % <u>Corrective Action:</u> Flag data associated with unacceptable MS. (See SOP CORP-MT-0003 for detailed corrective action procedure and for other QC procedures.)
	Matrix Spike Duplicate	200 series 1631E	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples. 1631E frequency is 1 in 10 samples, 71-125% RPD 24% <u>Criteria:</u> Recovery should be within 75-125 % , RPD⁽³⁾ should be within 20% <u>Corrective Action:</u> Flag data associated with unacceptable MSD SOP CORP-MT-0003	7000A series	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Recovery should be within 75-125 % , RPD⁽³⁾ should be within 20% <u>Corrective Action:</u> Flag data associated with unacceptable MSD SOP CORP-MT-0003

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	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Concentration less than reporting limit. Concentration less than reporting with the exception of lab common contaminants. Sample results <RL are also valid. <u>Corrective Action:</u> Rerun all samples associated with unacceptable blank SOP CORP-MT-0001	6010B 6020	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Concentration less than reporting limit. Concentration less than reporting with the exception of lab common contaminants. Sample results <RL are also valid. <u>Corrective Action:</u> Rerun all samples associated with unacceptable blank SOP CORP-MT-0001
	Laboratory Control Sample	200.7 200.8	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> percent recovery of analyte must be $\pm$ 85-115%. If LCS is biased high and samples are <RL, the results are valid. <u>Corrective Action:</u> Rerun all samples associated with unacceptable LCS SOP CORP-MT-0001	6010B 6020	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> percent recovery of analyte must be $\pm$ 20 %. If LCS is biased high and samples are <RL, the results are valid. <u>Corrective Action:</u> Rerun all samples associated with unacceptable LCS SOP CORP-MT-0001. 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 <u>Criteria:</u> Limits for percent recovery are 75-125% <u>Corrective Action:</u> Flag data associated with unacceptable Matrix Spike SOP CORP-MT-0001	6010B 6020	Frequency: 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Limits for percent recovery are 75-125% <u>Corrective Action:</u> Flag data associated with unacceptable Matrix Spike SOP CORP-MT-0001
	Matrix Spike Duplicate	200.7 200.8	Frequency: 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Limits for percent recovery are 75-125%, RPD ⁽³⁾ must be within 20% <u>Corrective Action:</u> Flag data associated with unacceptable Matrix Spike SOP CORP-MT-0001	6010B 6020	Frequency: 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Limits for percent recovery are 75-125%, RPD ⁽³⁾ must be within 20% <u>Corrective Action:</u> Flag data associated with unacceptable Matrix Spike SOP CORP-MT-0001
	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 <u>Criteria:</u> 10% difference. 10% difference only applied if sample results are >50 times IDL. <u>Corrective Action:</u> Flag data associated with unacceptable Serial Dilution SOP CORP-MT-0001	6010B 6020	Frequency: 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> 10% difference. 10% difference only applied if sample results are >50 times IDL. <u>Corrective Action:</u> Flag data associated with unacceptable Serial Dilution SOP CORP-MT-0001

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	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Concentration less than reporting limit <u>Corrective Action:</u> Rerun all samples associated with unacceptable blank	8021B	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Concentration less than reporting limit <u>Corrective Action:</u> Rerun all samples associated with unacceptable blank
	Laboratory Control Sample	602	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> percent recovery must be within acceptance limits given in method for each analyte <u>Corrective Action:</u> Rerun all samples associated with unacceptable LCS	8021B	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> percent recovery for each analyte must be within laboratory acceptance limits <u>Corrective Action:</u> Rerun all samples associated with unacceptable LCS
	Matrix Spike	602	<u>Frequency:</u> 1 per 10 samples from each site or 1 per month, whichever is more frequent <u>Criteria:</u> percent recovery for each analyte should be within advisory limits given in method <u>Corrective Action:</u> Flag data associated with unacceptable Matrix Spike	8021B	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> percent recovery for each analyte should be within laboratory acceptance limits <u>Corrective Action:</u> 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	<u>Frequency</u> : 1 with each batch of samples processed not to exceed 20 samples <u>Criteria</u> : percent recovery for each analyte should be within laboratory acceptance limits <u>Corrective Action</u> : 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) <u>Method Blank Criteria and LCS</u> : All surrogates must be within laboratory established control limits before sample analysis may proceed. <u>Sample Criteria</u> : Re-extract samples or flag sample data not meeting surrogate criteria	8021B	Surrogates spiked into method blank and all samples (QC included) <u>Method Blank Criteria and LCS</u> : All surrogates must be within laboratory established control limits before sample analysis may proceed. <u>Sample Criteria</u> : Reprep 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	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Concentration less than reporting limit <u>Corrective Action:</u> Rerun all samples associated with unacceptable blank
	Laboratory Control Sample	--	N/A	8021B	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> percent recovery for each analyte must be within laboratory acceptance limits <u>Corrective Action:</u> Rerun all samples associated with unacceptable LCS
	Matrix Spike	--	N/A	8021B	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> percent recovery for each analyte should be within laboratory acceptance limits <u>Corrective Action:</u> Flag data associated with unacceptable Matrix Spike
	Matrix Spike Duplicate	--	N/A	8021B	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> percent recovery for each analyte should be within laboratory acceptance limits <u>Corrective Action:</u> 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) <u>Method Blank Criteria and LCS:</u> All surrogates must be within laboratory established control limits before sample analysis may proceed. <u>Sample Criteria:</u> Reprep 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	<u>Frequency:</u> 1 with each extraction batch of samples not to exceed 20 samples <u>Criteria:</u> Percent recovery must be within acceptance limits given in method for each analyte <u>Corrective Action:</u> Re-extract all samples associated with unacceptable LCS	8151A	<u>Frequency:</u> 1 with each extraction batch of samples not to exceed 20 samples <u>Criteria:</u> Percent recovery for each analyte must be within laboratory acceptance limits <u>Corrective Action:</u> Re-extract and reanalyze all samples associated with unacceptable LCS
	Matrix Spike	615 ³	<u>Frequency:</u> 1 per 10 samples from each site or 1 per month, whichever is more frequent <u>Criteria:</u> Percent recovery for each analyte should be within advisory limits given in method <u>Corrective Action:</u> Flag data associated with unacceptable Matrix Spike	8151A	<u>Frequency:</u> 1 with each extraction batch of samples not to exceed 20 samples <u>Criteria:</u> Percent recovery for each analyte should be within laboratory acceptance limits <u>Corrective Action:</u> Flag data associated with unacceptable Matrix Spike
	Matrix Spike Duplicate	615 ³	N/A	8151A	<u>Frequency:</u> 1 with each extraction batch of samples not to exceed 20 samples <u>Criteria:</u> percent recovery for each analyte should be within laboratory control limits <u>Corrective Action:</u> 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) <u>Method Blank Criteria and LCS:</u> All surrogates must fall within laboratory established control limits before sample analysis may proceed. <u>Sample Criteria:</u> 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	<u>Frequency:</u> 1 with each extraction batch of samples not to exceed 20 samples <u>Criteria:</u> Concentration less than reporting limit <u>Corrective Action:</u> Rerun all samples associated with unacceptable blank	8081A 8082	<u>Frequency:</u> 1 with each extraction batch of samples not to exceed 20 samples <u>Criteria:</u> Concentration less than reporting limit <u>Corrective Action:</u> 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	<u>Frequency:</u> 1 with each extraction batch of samples not to exceed 20 samples <u>Criteria:</u> percent recovery must be within acceptance limits given in method for each analyte <u>Corrective Action:</u> Rerun all samples associated with unacceptable LCS	8081A 8082	<u>Frequency:</u> 1 with each extraction batch of samples not to exceed 20 samples <u>Criteria:</u> percent recovery for each analyte must be within laboratory acceptance limits <u>Corrective Action:</u> Rerun all samples associated with unacceptable LCS
	Matrix Spike	608	<u>Frequency:</u> 1 per 10 samples from each site or 1 per month, whichever is more frequent <u>Criteria:</u> percent recovery for each analyte should be within advisory limits given in method <u>Corrective Action:</u> Flag data associated with unacceptable Matrix Spike	8081A 8082	<u>Frequency:</u> 1 with each extraction batch of samples not to exceed 20 samples <u>Criteria:</u> percent recovery for each analyte should be within laboratory acceptance limits <u>Corrective Action:</u> 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 80882	<u>Frequency:</u> 1 with each extraction batch of samples not to exceed 20 samples <u>Criteria:</u> percent recovery for each analyte should be within laboratory acceptance limits <u>Corrective Action:</u> 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) <u>Method Blank Criteria and LCS:</u> Results must fall within laboratory established control limits <u>Sample Criteria:</u> Re-extract and reanalyze samples or flag sample data not meeting surrogate criteria
Petroleum Hydro- carbons	* Method Blank	1664A	<u>Frequency:</u> 1 with each preparation batch <u>Criteria:</u> Concentration must be less than the reporting limit <u>Corrective Action:</u> Rerun all samples associated with unacceptable blank	9071B	<u>Frequency:</u> 1 with each preparation batch <u>Criteria:</u> Concentration must be less than the reporting limit <u>Corrective Action:</u> 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 Hydrocarbons (Cont'd)	Laboratory Control Sample	1664A	<u>Frequency:</u> 1 with each analytical batch <u>Criteria:</u> Waters - See limits in SOP, NC-WC-0084 Soils - Percent recovery must be within laboratory control limits <u>Corrective Action:</u> Rerun all samples associated with unacceptable LCS	9071B	<u>Frequency:</u> 1 with each analytical batch <u>Criteria:</u> Waters - See limits in SOP, NC-WC-0084 Soils - Percent recovery must be within laboratory control limits <u>Corrective Action:</u> Rerun all samples associated with unacceptable LCS
	Matrix Spike	1664A	<u>Frequency:</u> 1 with every 10 samples per site <u>Criteria:</u> See percent recovery limits in SOP, NC-WC-0084 <u>Corrective Action:</u> See SOP, NC-WC-0084	9071B	<u>Frequency:</u> 1 with every 10 samples per site <u>Criteria:</u> See percent recovery limits in SOP, NC-WC-0084 <u>Corrective Action:</u> See SOP, NC-WC-0084
	Matrix Spike Duplicate	1664A	<u>Frequency:</u> 1 with every 20 samples per site <u>Criteria:</u> See percent recovery and RPD limits in SOP, NC-WC-0084 <u>Corrective Action:</u> See NC-WC-0084	9071B	<u>Frequency:</u> 1 with every 10 samples per site <u>Criteria:</u> See percent recovery and RPD limits in SOP, NC-WC-0084 <u>Corrective Action:</u> 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	<u>Frequency:</u> 1 with each extraction batch of samples not to exceed 20 samples <u>Criteria:</u> Concentration less than reporting limit <u>Corrective Action:</u> Rerun all samples associated with unacceptable blank	8021B	<u>Frequency:</u> 1 with each extraction batch of samples not to exceed 20 samples <u>Criteria:</u> Concentration less than reporting limit <u>Corrective Action:</u> Rerun all samples associated with unacceptable blank
	Laboratory Control Sample	601	<u>Frequency:</u> 1 with each extraction batch of samples not to exceed 20 samples <u>Criteria:</u> percent recovery must be within acceptance limits given in method for each analyte <u>Corrective Action:</u> Rerun all samples associated with unacceptable LCS	8021B	<u>Frequency:</u> 1 with each extraction batch of samples not to exceed 20 samples <u>Criteria:</u> percent recovery for each analyte must be within laboratory acceptance limits <u>Corrective Action:</u> Rerun all samples associated with unacceptable LCS
	Matrix Spike	601	<u>Frequency:</u> 1 per 10 samples from each site or 1 per month, whichever is more frequent <u>Criteria:</u> percent recovery for each analyte should be within advisory limits given in method <u>Corrective Action:</u> Flag data associated with unacceptable Matrix Spike	8021B	<u>Frequency:</u> 1 with each extraction batch of samples not to exceed 20 samples <u>Criteria:</u> percent recovery for each analyte should be within laboratory acceptance limits <u>Corrective Action:</u> 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	<u>Frequency</u> : 1 with each extraction batch of samples not to exceed 20 samples <u>Criteria</u> : percent recovery for each analyte should be within laboratory acceptance limits <u>Corrective Action</u> : 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) <u>Method Blank Criteria and LCS</u> : All surrogates must be within laboratory established control limits before sample analysis may proceed. <u>Sample Criteria</u> : Re-extract samples or flag sample data not meeting surrogate criteria	8021B	Surrogates spiked into method blank and all samples (QC included) <u>Method Blank Criteria and LCS</u> : All surrogates must be within laboratory established control limits before sample analysis may proceed. <u>Sample Criteria</u> : 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	<u>Frequency:</u> 1 with each extraction batch of samples not to exceed 20 samples <u>Criteria:</u> percent recovery for each analyte should be within advisory limits given in method <u>Corrective Action:</u> Flag data associated with unacceptable Matrix Spike	8270C	<u>Frequency:</u> 1 with each extraction batch of samples not to exceed 20 samples <u>Criteria:</u> percent recovery for each analyte should be within laboratory acceptance limits <u>Corrective Action:</u> Flag data associated with unacceptable Matrix Spike
	Matrix Spike Duplicate	625	N/A	8270C	<u>Frequency:</u> 1 with each extraction batch of samples not to exceed 20 samples <u>Criteria:</u> percent recovery for each analyte should be within laboratory acceptance limits <u>Corrective Action:</u> 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) <u>Method Blank and LCS Criteria:</u> All surrogates must be in control before sample analysis may proceed. One surrogate per fraction may exceed control limits if greater than 10% recovery. <u>Sample Criteria:</u> Re-extract samples or flag sample data not meeting surrogate criteria	8270C	Surrogates spiked into method blank and all samples (QC included) <u>Method Blank and LCS Criteria:</u> All surrogates must be in control before sample analysis may proceed. One surrogate per fraction may exceed control limits if greater than 10% recovery. <u>Sample Criteria:</u> Re-extract and reanalyze samples or flag sample data not meeting surrogate criteria
	Internal Standards	625	<u>Frequency:</u> Internal standards spiked into method blank and all samples (QC included) <u>Criteria:</u> All internal standard recoveries must be within laboratory control limits <u>Corrective Action:</u> 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	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Concentration less than reporting limit <u>Corrective Action:</u> Rerun all samples associated with unacceptable blank	8260B	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Concentration less than reporting limit <u>Corrective Action:</u> Rerun all samples associated with unacceptable blank
	Laboratory Control Sample	624	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> percent recovery for each analyte should be within advisory limits given in method <u>Corrective Action:</u> Flag data associated with unacceptable Matrix Spike	8260B	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> percent recovery for each analyte must be within laboratory acceptance limits <u>Corrective Action:</u> 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	<u>Frequency:</u> 1 per ≤ 20 samples from each site or 1 per month, whichever is more frequent <u>Criteria:</u> percent recovery for each analyte should be within advisory limits given in method <u>Corrective Action:</u> Flag data associated with unacceptable Matrix Spike	8260B	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> percent recovery for each analyte should be within laboratory acceptance limits <u>Corrective Action:</u> Flag data associated with unacceptable Matrix Spike
	Matrix Spike Duplicate	624	N/A	8260B	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> percent recovery for each analyte should be within laboratory acceptance limits <u>Corrective Action:</u> 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) <u>Method Blank Criteria:</u> All surrogates must be in control before sample analysis may proceed. <u>Sample Criteria:</u> Re-extract samples or flag sample data not meeting surrogate criteria	8260B	Surrogates spiked into Method Blank and all samples (QC included) <u>Method Blank Criteria and LCS:</u> All surrogates must be in control before sample analysis may proceed. <u>Sample Criteria:</u> Re-extract and reanalyze samples or flag sample data not meeting surrogate criteria
	Internal Standards	624	<u>Frequency:</u> Internal standards spiked into method blank and all samples (QC included) <u>Criteria:</u> All internal standard recoveries must be within laboratory control limits <u>Corrective Action:</u> 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

SECTION 14.0

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 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 our TestAmerica North Canton commitment to the Quality Assurance (QA) 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 Quality Assurance 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 review, 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 shall 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 will provide 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 CA-Q-S-003, Management of Change.

SECTION 15.0

CONTROL OF RECORDS (NELAC 5.4.12)

TestAmerica North Canton 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 as described in SOP NC-QA-0019, Records Information Management.

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) Manager 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 15-1. Record and Retention Schedule

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

Type of Record	Retention	Disposition
LQM, policies, & SOPs	5 yrs from date of archiving	Shred or burn
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

All records are legible and 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. Records are maintained for a minimum of five years unless other wise specified by a client or regulatory requirement.

For raw data and project records, record retention shall 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 15-2 have lengthier retention requirements and are subject to the requirements in Section 15.1.3. Policy CW-L-P-001, Record Retention, provides additional information on record retention requirements.

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 15-3 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.

Table 15-2. Special Record Retention Requirements

Program	Retention Requirement
Ohio – Drinking Water	10 years
Michigan Department of Environmental Quality – all environmental data	10 years
Navy Facilities Engineering Service Center (NFESC)	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

15.1.2 All records are held secure and in confidence. Records maintained at the laboratory are located in the Records Storage area of the Warehouse. 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 hardcopy 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.

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. (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 shall 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-0019, 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 the LIMS for each method as required.
- Changes to hardcopy records shall follow the procedures outlined in Section 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 as long as the scanning process can 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 prior to the destruction of the hard copy which was scanned.
- 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 shall contain sufficient information to enable the analysis to be repeated under conditions as close as possible to the original. The records shall include the identity of laboratory personnel responsible for sample preparation, performance of each analysis and checking 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 shall follow the procedures outlined in Sections 13 and 20. 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 (previous discussions relate where most of this information is maintained – specifics may be added below):

- Laboratory sample ID code
- Date of analysis and time of analysis is 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 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
- 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.

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 hardcopy 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 to the accrediting body 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 hardcopy, write-protected backup copies, or an electronic audit trail controlling access.

15.5.4 TestAmerica North Canton has a record management system for control of instrument logbooks, standards logbooks, and records for data reduction, validation, storage, and reporting. Benchsheets are filed sequentially per method.

15.5.5 Records are considered archived when moved to Records Storage. 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. All records shall be 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. Access to the data is limited to laboratory and company employees.

15.5.6 In the event that the laboratory transfers ownership or goes out of business, TestAmerica North Canton 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.

15.5.7 Records Disposal

15.5.7.1 Records are removed from the archive and disposed 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.

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. Refer to Policy CW-L-P-001, Records Retention.

SECTION 16

AUDITS (NELAC 5.4.13)

16.1 OVERVIEW

Audits measure laboratory performance and insure compliance with accreditation/certification and project requirements. Audits specifically provide management with an on-going assessment of the quality of results produced by the laboratory, including how well the policies and procedures of the QA system and the Ethics and Data Integrity Program are being executed. They are also instrumental in identifying areas where improvement in the QA system will increase the reliability of data. There are two principle types of audits: Internal and External. Internal audits are performed by laboratory or corporate personnel. External audits are conducted by regulators, clients or third-party auditing firms. In either case, the assessment to program requirements is the focus.

Table 16-1. Audit Types and Frequency

Internal Audits	Description	Performed by	Frequency
	Analyst & Method Compliance	QA Department or Designee	- 100% of all methods over a two year period. - 100% of all analysts annually.
	Instrument	QA Department or Designee	100% of all organic instruments and any inorganic chromatography instruments. Annually.
	Work Order/ Final Report	QA Department or Designee	- 1 complete report each month.
	Support Systems	QA Department or Designee	- Annual for entire labs support departments & equipment (e.g., thermometers, balances), can be divided into sub-sections over the course of the year.
	Performance Audits (Double-Blind PTs)	Corporate QA, Laboratory QA Department or Designee	- As needed.
	Special	QA Department or Designee	- As Needed
External Audits	Description	Performed by	Frequency
	Program / Method Compliance	Regulatory Agencies, Clients, accreditation organizations	- As required by program and/or clients needs
	Performance Audits	Provided by a third party.	- As required by a client or regulatory agency. Generally provided semi-annually through the analysis of PT samples.

16.2 INTERNAL AUDITS

Annually, the laboratory prepares a schedule of internal audits to be performed throughout the year. As previously stated, these audits verify and monitor that operations continue to comply with the requirements of the laboratory's QA Manual and the Corporate Ethics Program. A

schedule of internal audits is maintained by the QA Manager in the *Internal Audit Workbook*. An example can be found in Figure 16-1.

It is the responsibility of the QA Manager to plan and organize audits in consideration of the laboratory workload and the department personnel schedules so that all pertinent personnel and operations are thoroughly reviewed. When designees (other than QA Department personnel and approved by the QA Manager) perform audits, the QA Manager shall ensure that these persons do not audit their own activities, except when it can be demonstrated that an effective audit will be carried out. In general, the auditor:

- Is neither the person responsible for the process being audited nor the immediate supervisor of the person responsible for the project/process.
- Is free of any conflicts of interest.
- Is free from bias and influences that could affect objectivity.

Laboratory personnel (e.g., supervisors and analysts) may assist with both method and support system audits as long as the items listed in the above paragraph are observed. These audits are conducted according to defined criteria listed in the checklists of the *Internal Audit Workbook*. These personnel must be approved by the QA Manager; and must complete the audit checklists in their entirety. This process introduces analyst experience and insight into the laboratory's auditing program.

The auditor must review the previous audit report and identify all items for verification of corrective actions. A primary focus will be dedicated to the ability of the laboratory to correct root-cause deficiencies and that the corrective action has been implemented and sustained as documented.

16.2.1 Systems

An annual 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. This audit is performed in portions throughout the year through method, analyst, instrument, Work Order/Final Report, and support system audits. Audits are documented and reported to management within one week of their performance. Systems audits cover all departments of the facility--both operational and support. The multiple audits are compiled into one systems audit package at the end of the year (*Internal Audit Workbook*).

16.2.1.1 Method, Analyst, Instrument and Work Order/Final Report Audits

Procedures for the method compliance, analyst, instrument and work order/final report audits are incorporated by reference to SOP CA-Q-S-004, Method Compliance and Data Authenticity Audits. These audits are not mutually exclusive. For example, the performance of a method audit will also cover multiple analysts and instruments. The laboratory's goal is to annually review all analysts and instruments as described in SOP CA-Q-S-004. The laboratory will also audit all methods within a two-year time period, and audit a minimum of one Work Order/Final Report from receiving through reporting on a monthly basis.

16.2.1.2 Support Systems

Support system audits are performed to ensure that all departments & ancillary equipment are operating according to prescribed criteria. Support system audits include the review of both non-analytical and operational departments. Support equipment audits, e.g., metrology items, include the review of balance calibrations, weight calibrations; water quality testing, etc.. Non-analytical may include sample receiving and bottle preparation. These types of support audits ensure that the operations are being performed to support ethical data as well as ensuring the accuracy & precision of the utilized equipment.

These audits can be performed in portions throughout the year or in one scheduled session. However, the audit schedule must document that these aspects are reviewed annually. Many of the metrology systems are considered to be surveillance activities that can be monitored by QA personnel or delegated to specified department personnel. These surveillance activities are performed on a semi-annual basis unless issues warrant a greater frequency or previous audits continually showing no deficiencies allow the frequency to be reduced to once a year.

An example audit checklist can be found in Figure 16-2. Instructions for reporting findings are included in the *Internal Audit Workbook*. In general, findings are reported to management within 1 week of the audit and a response is due from management within 30 days.

16.2.2 Performance Audits

Corporate QA may arrange for double blind PT studies to be performed in the laboratories. Results are given to Management and Corrective actions of any findings are coordinated at each facility by the QA Managers and Laboratory Directors/Managers. These studies are performed on an as needed basis. They may be performed when concerns are raised regarding the performance of a particular method in specific laboratories, periodically to evaluate methods that may not normally be covered in the external PT program or may be used in the process of developing best practices. The local QA Manager may also arrange for PT studies on an as needed basis (refer to Section 16.3.2 for additional information on Performance Audits).

16.2.3 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.

16.3 EXTERNAL AUDITS

TestAmerica facilities are routinely audited by clients and external regulatory authorities. External audits are performed when certifying agencies or clients conduct on-site inspections or submit performance testing samples for analysis. It is the TestAmerica 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. The Laboratory 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. This time frame is generally 30 days.

Be aware that NELAC requires that the audit response report be acceptable to the primary accrediting authority after the second submittal. The lab shall have accreditation revoked for all or any portion of its scope of an accreditation for any or all fields of testing, a method, or analyte within a field of testing if it is not corrected.

TestAmerica North Canton cooperates with clients and their representatives to monitor laboratory 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.

16.3.1 Confidential Business Information (CBI) Considerations

During on-site audits, on-site 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.

16.3.2 Performance Audits

The laboratory is involved in performance audits conducted as directed by state agencies through the analysis of PT samples provided by a third party. The laboratory generally participates in the following types of PT studies—water pollution and soil studies.

- It is TestAmerica's policy that PT samples be treated as typical samples in the production process. Further, 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 manufacturer's instructions. Holding times should 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 will obtain the COC information from the documentation provided with the PTs with review by QA or other designated staff.

- Vials will be prepared as required in the instruction set provided with the samples. After preparation to full volume the sample may be spiked, digested, concentrated, etc., as would be done for any normal sample requiring similar analysis.
- PT samples will 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. An example of this is 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 shall be the same as with routine environmental samples.
- Instructions may be included in the laboratory SOPs on 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 may be used. Results below the routine reporting limit are reviewed against PT instructions and analyte PT reporting limit values.
- No special reviews shall be performed by operation 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.

16.4 AUDIT FINDINGS

Internal or External Audit findings should be documented using the corrective action process. The laboratory is expected to prepare a response to audit findings within 30 days of receipt of an audit report unless the report specifies a different time frame. The response may include action plans that could not be completed within the 30-day timeframe. In these instances, a completion date must be set and agreed to by Operations Management and the QA Manager.

Responsibility for developing and implementing corrective actions to findings is the responsibility of the Group Leader where the finding originated. Findings that are not corrected by specified due dates are reported monthly to management in the QA monthly report.

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.

The procedures must be in accordance to SOP CA-L-S-001, Internal Investigations of Data Discrepancies and Determination of Data Recall.

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.

Figure 16-1.

Example - Internal Audit Workbook

				TestAmerica <Name> Last Updated: 9/10/2007		
THE LEADER IN ENVIRONMENTAL TESTING				Workbook Instruction No. CA-Q-WI-011		
Internal Audit Workbook Summary Page						
Note: Click on the (Summary Page) to located on each audit sheet to return to this page.						
* The lab may choose to audit these areas with each method/analyst/instrument audit. The auditor must document on the checklist that this item is audited as part of the <defined> audit.						
	Area Audited	Audit Type	Audit Cycle	Scheduled	Date Audited	Date Closed
	(Click on the Area to get to that Spreadsheet)					
1	Balances	System	6 mo			
2	Temperature Logs/Thermometers	System	6 mo			
3	Sample Storage and Disposal	System	1 yr			
4	Maintenance Logs *	System	6 mo			
5	Volatile Storage Blanks	System	6 mo			
6	Lab Water Quality Testing	System	6 mo			
7	Sample Log In	System	1 yr			
8	Shipping Procedures	System	1 yr			
9	Computer Operations (LIMS)	System	1 yr			Pending Corp. IT Policies
10	SOP & Document Distribution System	System	1 yr			
11	Archiving Electronic & Paper Records	System	1 yr			Pending Corp. IT Policies
12	Statistical Process Control	System	1 yr			
13	Data Review System	System	1 yr			
14	Final Report Generation	System	1 yr			
15	Standards/Reagents *	System	6 mo			
16	Manual Integration *	System	1 yr			
17	Corrective Action System	System	1 yr			
18	Training Records	System	6 mo			
19	MDLs	System	1 yr			
20	SOPs - Prep/Review/Update Process	System	1 yr			
21	Purchasing/Procurement	System	1 yr			
22	Eppendorf/Diluter/Dispenser Calibration Check	System	6 mo			
23	Subcontract Lab Approval	System	1 yr			
24	Customer Complaint System	System	1 yr			
25	Methods	Method	2 yr			
						
						
						

Figure 16-2.

Example – Internal Audit System Checklist: Corrective Actions



(Summary Page)

TestAmerica <Location>

INTERNAL AUDIT - Corrective Actions

[Printed Name(s) or Date(s)]

Area Audited: _____
Auditor: _____
Date: _____
Persons Contacted During Audit: _____
Date Reported to Department Manager: _____
Reported To: _____
Date Reported to Lab Director/Manager: _____
Reported To: _____
Date Response Due: _____
Response Received and Accepted by QA Manager: _____
Associated Corrective Action Report Number(s): _____
Scheduled Follow-up: _____

Item	Requirement	Ref.	Y	N	NA	Evidence/Comments	Follow Up
1	Does the laboratory have a corrective action program in place?	5.4.10.1					
2	Does the laboratory have a current corrective action SOP or is this information in the QA Manual?	5.4.10.1					
3	Do all laboratory personnel have documented training and access to initiate corrective actions?	5.4.10.1					
4	Are causes clearly identified by department, staff name, scope of issue (how many reports affected)?	5.4.10.6					
5	Is a root cause for the issue identified?	5.4.10.2					
6	Is a corrective action (plan) clearly described?						
7	Was the corrective action fully implemented?						
8	Is documentation (if applicable) completed as specified by the corrective action (training, revised SOP, etc)						
9	Has a follow-up assessment been conducted to verify the corrective action was successful?						
10	Are corrective actions reviewed on a regular basis by management?	5.4.10.6a 5					
11	Is there a defined distribution flow for corrective action notification, review, closure, and follow-up?	5.4.10.6a					
12	Are non-conformances reviewed on a regular basis and used, if necessary, to initiate root cause corrective actions?						
13	Does the lab have a documented procedure for QC corrective action (i.e., documented within each method / parameter SOP or in the QA Manual)?	4.10.1					
14	Verify Corrective Actions from previous systems audits. List Items:						
15							
16							
17							

Auditor Signature: _____

Primary Reference(s): Corporate SOP CA-Q-S-002, Acceptable Manual Integration Practices
NELAC Standard, June 2003
DoD Quality Systems Manual, Version 3, January 2006
EPA Manual for the Certification of Laboratories Analyzing Drinking Water

SECTION 17

MANAGEMENT REVIEWS (NELAC 5.4.14)

17.1 QUALITY ASSURANCE REPORT

A comprehensive QA Report shall be prepared each month by the laboratory's QA Department and forwarded to the Laboratory Director/Manager for review and comments. The final report shall be submitted to the Laboratory Director as well as the appropriate Quality Director and General Manager. All aspects of the QA system are reviewed to evaluate the suitability of policies and procedures. At a minimum, the report content will contain the items listed below. 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.

The TestAmerica QA Report template is comprised of a discussion of three key QA issues facing the laboratory and ten specific sections (Figure 17-1):

- **Metrics:** Describe actions or improvement activities underway to address any outlying quality metrics that have been reported in the monthly Quality System Metrics Table.
- **Quality System Metrics Table:** The report also includes statistical results that are used to assess the effectiveness of the quality system. Effective quality systems are the responsibility of the entire laboratory staff. Each laboratory provides their results in a template provided by Corporate QA (Figure 17-2).
- **SOPs:** Report SOPs that have been finalized and report status of any outstanding SOP reviews.
- **Corrective Actions:** Describe highlights and the most frequent cause for report revisions and corrective/preventive action measures underway. Include a discussion of any recalls handled at the lab level as per Section 6.2.2 in the Investigation/Recall SOP, CA-L-S-001. Include a section for client feedback and complaints. Include both positive and negative feedback. Describe the most serious client complaints and resolutions in progress.
- **MDLs and Control Limits:** Report which MDLs/ MDL verifications are due. Report the same for Control Limits.
- **Audits:** Report Internal and External Audits that were conducted. Include all relevant information such as which methods, by whom, corrective actions needed by when and discuss unresolved audit findings.
- **Performance Testing (PT) Samples:** Report the PT tests that are currently being tested with their due dates, report recent PT results by study, acceptable, total reported and the month and year.
- **Certifications:** Report on any certification programs being worked on by due date, packages completed. Describe any issues, lapses, or potential revocations.
- **Regulatory Updates:** Include information on new state or federal regulations that may impact the laboratory. Report new methods that require new instrumentation, deletion of methods, changes in sampling requirements and frequencies etc...

- **Miscellaneous:** Include any issues that may impact quality within the laboratory.
- **Next Month:** Report on plans for the upcoming month.
- **Lab Director Comments Section:** This section gives the Laboratory Director/Manager the opportunity to comment on issues discussed in the report and to document plans to resolve these issues. Unresolved issues that reappear in subsequent monthly reports must be commented on by the Laboratory Director/Manager.
- **Quality System Metrics Table:** The report also includes statistical results that are used to assess the effectiveness of the quality system. Effective quality systems are the responsibility of the entire laboratory staff. Each laboratory provides their results in a template provided by Corporate QA (Figure 17-2).

On a monthly basis, Corporate QA compiles information from all the monthly laboratory reports. The VP-QA/EHS prepares 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 Analytical Division Senior Management Team and General Managers.

17.2 ANNUAL MANAGEMENT REVIEW

The Senior Lab Management Team (Laboratory Director, Technical Director, Operations Manager, QA Manager, HR Supervisor, I.T. Supervisor) 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. 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 will summarize any critical findings that cannot be solved by the lab and report them to Corporate IT.

This review uses information generated during the preceding year to assess the “big picture” by ensuring that routine quality actions taken and reviewed on a monthly basis are not components of larger systematic concerns. The monthly review (refer to Section 17.1) 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 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.

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 the 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 shall be followed (SOP 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.

The Chairman/CEO, President/CEO, COOs, and Quality Directors receive a monthly report from the VP of Quality and EHS summarizing any current data integrity or data recall investigations as described in SOP CA-L-S-001. The General Managers are also made aware of progress on these issues for their specific labs.

Figure 17-1.

Example - QA Monthly Report to Management

LABORATORY: x
PERIOD COVERED: Month/Year
PREPARED BY: x DATE: Month Day, Year
DISTRIBUTED TO: xx (Include LD, GM, QA Director, etc...)

THREE KEY ISSUES FOR MONTH:

Include a discussion of three key issues that were focused in on this month.

1. x
 2. x
 3. x
-

1. METRICS

Describe actions or improvement activities underway to address any outlying quality metrics.

2. SOPs

See Tab for SOP specifics.

The following SOPs were finalized (or reviewed for accuracy): (See Tab)

The following SOPs are due to QA: xx

In QA to complete: xx

3. CORRECTIVE ACTION

Highlights: xx

Revised Reports:

Describe the most frequent cause for report revisions and corrective/preventive action measures underway.

Data Investigations/Recalls (Corporate Data Investigation/Recall SOP) :

Include a discussion of any recalls handled at the lab level as Corp SOP.

Client Feedback and Complaints:

Include both positive and negative feedback.

Describe the most serious client complaints) and resolutions in progress.

4. MDLs AND CONTROL LIMITS

MDLs Due:

Control Limits Due:

5. AUDITS

INTERNAL AUDITS

Discuss Any Outstanding Issues (or Attach Summary):

EXTERNAL AUDITS

Discuss Any Outstanding Issues (or Attach Summary):

6. PT SAMPLES

The following PT samples are now in house (Due Dates):

xx

7. CERTIFICATIONS

Certification Packages Being Worked On (Include Due Date):

x

Describe any issues, lapses, or potential revocations.

8. REGULATORY UPDATE

Include information on new state or federal regulations that may impact the laboratory – new methods that require new instrumentation, deletion of methods, changes in sampling requirements or frequencies, ...

9. MISCELLANEOUS

Include any issues that may impact quality within the laboratory.

10. NEXT MONTH

Items planned for next month.

LAB DIRECTOR COMMENTS AND PLANNED CORRECTIVE ACTIONS:

LAB DIRECTOR REVIEW:

DATE:

Figure 17-2.

Example - Laboratory Metrics Categories

No. of reports for month
No. of reports revised due to lab error
Percent revised reports
No. of Data Recall Investigations
No. of reports actually recalled
No. of Corrective Action Reports
No. of Corrective Action Reports still open
Total number of unresolved open Corrective Action Reports
Percent of unresolved open Corrective Action Reports
No. of reports independent QA reviewed
Percent QA Data Review: Reports
No. of technical staff (analysts/technicians, including temps)
No. of analyst work product reviewed year-to-date
No. of analytical instruments with electronic data file storage capability
No. of Analytical instruments reviewed for data authenticity year-to-date
Percent Analyst/Instrument Data Authenticity Audits
No. of client complaints
No. of client compliments
No. of planned internal audits
No. of planned internal method audits performed year-to-date
Percent annual Internal Audits complete
No. of open Internal Audit findings past due
Total Number of External Audit findings
No. of open External Audit findings past due
Percent External Audit findings past due
No. of PT analytes participated and received scores
No. of PT analytes not acceptable
Percent PT cumulative score
No. of PT repeat analyte failures cumulative (analyte failed more than once in 4 consecutive studies by PT type--only applies to failed analytes)
No. of SOPs

No. of SOPs reviewed/revised within 24 months
No. of methods or administrative procedures without approved SOPs
SOP status
Method certification losses due to performance/audit issues
Hold Time violations due to lab error
Date of last Comprehensive Ethics Training session
No. of staff that haven't received Comprehensive Ethics Training (>90 days from employment date)
MDL status (good, fair, or poor) >90%, >70%, <70%
Training Documentation Records (good, fair, or poor)
LQM Revision/review date
QAM updated to new integrated template
Last annual Internal Audit date (opened, closed)
Last Management QS Review date
No. of SOPs required for 12-month review cycle (DOD or drinking water)
No. of SOPs for 12-month cycle/revised within 12 months (<i>Includes QS and Methods Listed in QSM</i>)
12-month percent SOP status (<i>Includes QS and Methods Listed in QSM</i>)

SECTION 18

PERSONNEL (NELAC 5.5.2)

18.1 OVERVIEW

TestAmerica'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 that is undergoing training shall have appropriate supervision until they have demonstrated their ability to perform their job function on their own. Staff shall 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 shall 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

TestAmerica 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. 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)
GFAA, 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 1 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 – <u>General</u>	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 – <u>Wet Chem</u> 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

Specialty	Education	Experience
Technical Director - Microbiology	Bachelors degree in applied science with at least 16 semester hours in general microbiology and biology An advanced (MS, PhD.) degree may substitute for one year of experience	And 2 years of 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.

18.3 **TRAINING**

TestAmerica 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	Day of hire	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 20.

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 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 & records; benefit programs; timekeeping/payroll; and employee conduct, e.g., ethics. This information is maintained in the employee's secured personnel file.

Further details of the laboratory's training program are described in the Laboratory Training SOP CORP-QA-0013, Employee Orientation and Training.

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, 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 CA-L-P-001 and an Ethics Statement/Agreement (Appendix 1). 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 (Appendix 1)
- 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 19

ACCOMMODATIONS AND ENVIRONMENTAL CONDITIONS (NELAC 5.5.3)

19.1 OVERVIEW

TestAmerica North Canton is an 11,466 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.

19.2 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 will be discontinued until the environmental conditions are returned to the required levels (refer to Section 12).

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. 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.

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.

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 TestAmerica North Canton. 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 20.0

TEST METHODS AND METHOD VALIDATION (NELAC 5.5.4)

20.1 OVERVIEW

TestAmerica North Canton 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 North Canton 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 preparation, review, revision, and control are incorporated by reference to SOP CW-Q-S-002, Writing a Standard Operating Procedure (SOP), and SOP NC-QA-0027, 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.

20.3 LABORATORY METHODS MANUAL

For each test method, the laboratory shall have available the published referenced method as well as the laboratory developed SOP. Refer to the corporate SOP CW-Q-S-002, Writing a Standard Operating Procedure, and SOP NC-QA-0027, Preparation and Management of Standard Operating Procedures, for content and requirements of technical and non-technical SOPs.

Note: If more stringent standards or requirements are included in a mandated test method or regulation than those specified in this manual, the laboratory shall 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.

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 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.

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 shall be used.

In general, TestAmerica North Canton follows procedures from the referenced methods shown below in Section 20.3.1.4.

When clients do not specify the method to be used or methods are not required, the methods used will 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:

- 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; 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 20-1. 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)	--

Analytical Parameters	Matrix	Fields of Testing			
			CWA	RCRA (SW846)	Other
Chromium, Hexavalent	Water		EPA 3500-Cr-D	EPA 7196A	--
	Waste		EPA 3500-Cr-D	EPA 7196A	--
	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	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 -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 20-2. 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 20-3. Methods for Mercury by Cold Vapor Atomic Fluorescence

Analytical Parameters	Matrix	Fields of Testing			
			CWA	RCRA (SW846)	Other
Mercury, Low Level (CVAFS)	Water		EPA 245.7	--	EPA 1631E

TABLE 20-4. 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 20-5. 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 245.7	---	EPA 1631E

TABLE 20-6. 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 20-7. 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 shall 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 will be documented.

20.4.2 Demonstration of Capability

Before the laboratory may institute a new method and begin reporting results, the laboratory shall 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.
- 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 laboratories archiving procedures. Refer to Section 15, Control of Records.
- 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).

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).
- The reporting limit is set at or above the first standard of the curve for the analyte.
- The client request is documented and the lab informs the client of its procedure for working with unusual compounds. This must be addressed in the Case Narrative.

- Refer to Section 12, Control of Non-Conforming Work.

20.4.3 Initial Demonstration of Capability (IDOC) Procedures

- 20.4.3.1 At least four aliquots shall be prepared (including any applicable clean-up procedures) and analyzed according to the test method (either concurrently or over a period of days).
- 20.4.3.2 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 CORP-QA-0013, Employee Orientation and Training, for details on this procedure.

A certification statement (see Figure 20-1 as an example) shall 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.

20.5 LABORATORY DEVELOPED METHODS AND NON-STANDARD METHODS

Any new method developed by the laboratory must be fully defined in an SOP/Methods Manual (Section 20.2) 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. The information included in the checklist below (Figure 20-2) is needed before samples are accepted for analysis by a new 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 (from 2003 NELAC Standard).

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.

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 shall be followed. The laboratory determinations of MDLs are described in Section 20.6.

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 concluded. The QL is the minimum level at which both the presence of an analyte and its concentration can be reliably determined. 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.

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, a determination of the applicable range of the method may be performed. In most cases, range is determined and demonstrated by comparison of the response of an analyte in a curve to established or targeted criteria. The curve is used to establish the range of quantitation and the lower and upper values of the curve represent upper and lower quantitation limits. Curves are not limited to linear relationships.

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 Section 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 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.

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 will use 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. 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 ensure 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 The Reporting Limit should generally be between two and five 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 labs, the RL is $\geq 3x$ MDL. If a sample is diluted, the reported MDL is adjusted according to the dilution factor.

20.7.5 If the MDL is $< 1/10$ of the spike concentration for more than 10% of the analytes in the method ($< 1/5$ of spike recovered for DoD for water samples), the MDL must be repeated (including extraction or digestion) using a lower spike level unless the percent recovery is $< 50\%$ or $> 150\%$ of the “true value”. Note: The concentration of the spike will be at a level below the calibration range. Note: The spiking concentration must be less than the reporting limit.

20.7.6 The calculated MDL cannot be not greater than the spike amount.

20.7.7 If the most recent 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 (20.7), 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.).

20.7.8 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.

20.7.9 The initial MDL is calculated as follows:

$$\text{MDL} = t_{(n-1, 1-a = 0.99)} \times (\text{Standard Deviation of replicates})$$

where $t_{(n-1, 1-a = 0.99)} = 3.143$ for seven replicates.

20.7.10 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.). The procedures utilized must be documented in the MDL SOP NC-QA-0021, Evaluation of Method Detection Limits for Chemical Tests.

20.7.11 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, will be qualified as an estimated value.

20.7.12 Detections reported down to the MDL must be qualitatively identified.

20.7.13 MDLs and Reporting limits are adjusted in LIMs based on moisture content and sample aliquot size.

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 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.

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 two times the calculated MDL. The analytes must be qualitatively identified or see Section 20.6.7 for other options. This verification does not apply to methods that are not readily spiked, e.g., pH, turbidity, etc. If the MDL does not verify, then the lab will not report to the MDL, or redevelop their MDL or use the level where qualitative identification is established (see Section 20.6.7). MDLs must be verified at least quarterly.

20.9.2 When a Reporting limit is established, it must be initially verified by the analysis of a low-level standard or QC sample (LCS at 1-2 the reporting limit) and annually thereafter. Unless there are requirements to the contrary, the acceptance criteria is $\pm 50\%$. The annual requirement is waived for methods that have an annually verified MDL.

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 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.

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 will not be identified by the computer as a positive match of the analyte of interest.

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 will not be 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, atomic absorption, or fluorescence profiles.

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 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). 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.

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. 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.

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.

- Changes to reports are documented via Change Order Forms and by noting "Revised" on the cover page.
- 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 will 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 shall be installed on all servers and workstations. The anti-virus software shall be configured to check for virus signature file and program updates on a daily basis and these updates will be pushed to all servers and workstations. The anti-virus software will be configured to clean any virus-infected file if possible, otherwise the file will be deleted. Disks and CDs brought from any outside source that are not OEM software must be scanned for viruses before being accessed.
- **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 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 laboratories 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 QA staff before adding to standard reference tables.

(g) Please contact Corporate QA or IT staff if you have any questions regarding implementation or interpretation of this policy.

20.13.1.2 **Ensure Information Availability:** Protection against loss of information or service through scheduled back-ups, 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 file servers.
- 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 five years from the last raw data generated using that software. Length of time is dependent on local regulations or client requirements, e.g., OVAP requires ten years.
- System Back-up Overview and Procedures
 - Data from both servers and instrument attached PC's 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 will be 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, and encryption 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.
- The company website contains SSL (Secure Socket Layer) encryption for secure website sessions and data transfers.
- Electronic documents such as PDF files and electronic data deliverables may be made available to clients via the secure web site. The logon page for this web site contains an agreement that the customer must accept before they will be logged on which states that the customer agrees not to alter any electronic data made available to them.

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.

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.

20.13.2.1 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.

20.13.2.2 In general, concentration results are reported in milligrams per liter (mg/l) or micrograms per liter ($\mu\text{g/l}$) for liquids and milligrams per kilogram (mg/kg) or micrograms per kilogram ($\mu\text{g/kg}$) for solids. The units “mg/l” and “mg/kg” are the same as “parts per million (ppm)”. The units “ $\mu\text{g/l}$ ” and “ $\mu\text{g/kg}$ ” 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 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.

20.13.2.4 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.

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.)

- 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 QA Manager at the facility. The QA Manager controls all worksheets following the procedures in Section 6.

20.13.4 Review / Verification Procedures

20.13.4.1 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 T-Q-005, Recording Laboratory Observations and Raw Data.

20.13.4.2 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.

20.13.4.2.1 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 shall 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 S-Q-004, Acceptable Manual Integration Practices.

For data that are input by an analyst and processed using a computer, a copy of the input shall 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.

20.13.4.2.2. 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, supervisor, 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.

20.13.4.2.3. 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 will be finalized by the PM. This narrative will include relevant comments collected during the earlier reviews.

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 shall 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.
- 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 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 20-1.
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 20-2.

Example - New Method / Additional Analyte Checklist

New Method / Additional Analyte Checklist

The following items are **required** to be completed prior to the acceptance of client samples.

Fill in any blanks that do not apply with "NA".

Forward to Analytical Group, Operations Manager, Technical Director, and QA.

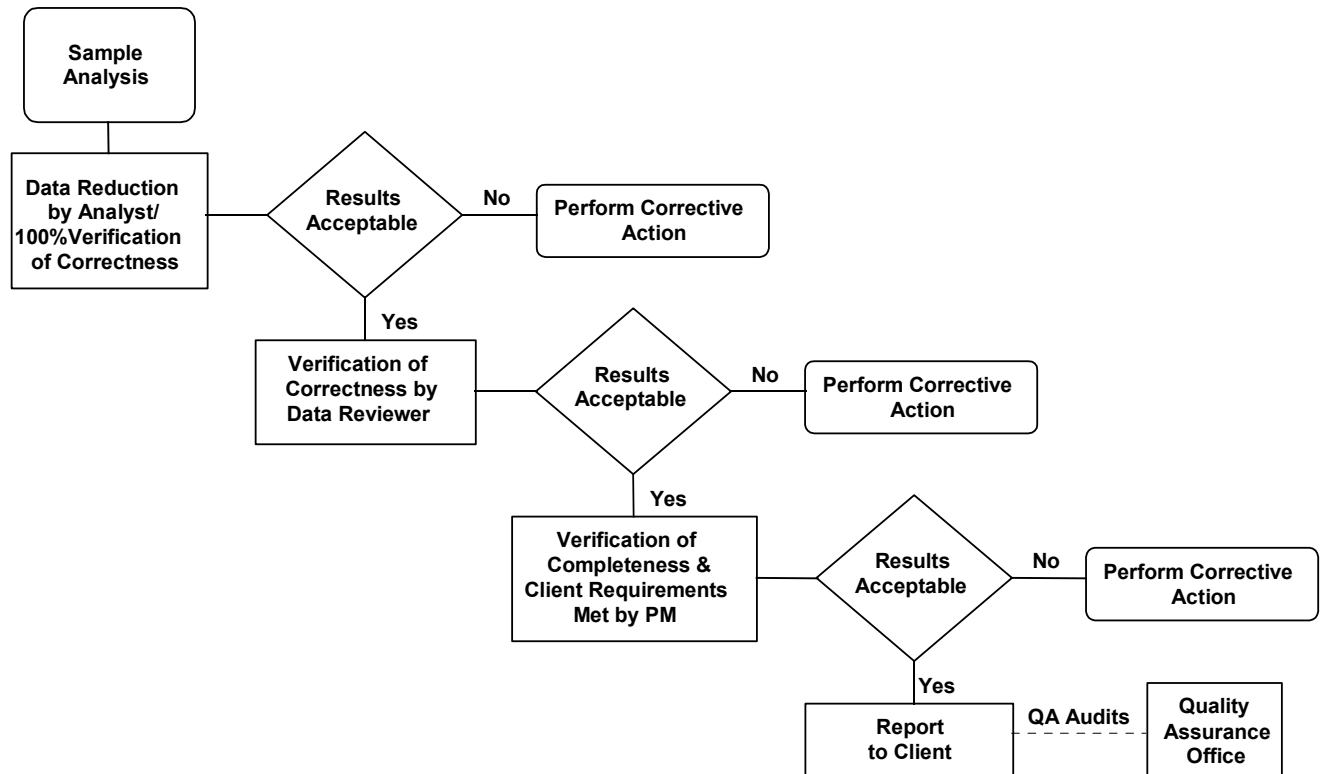
New Method – include method number and reference _____

New Analytes – list analyte and reporting limit

Matrix: water – solid – waste	
Analytical Groups Contacted	
Standard available for new analyte(s), including second source. If not, include time frame for order.	
Cost	
MDL study required	
MDL study completed	
LCS/MS/MSD spike required	
Project tied to QAPP? Lab has a copy?	
Project Program: DoD QSM, LCG, Client specific, State Specific	
Special QC Requirements: control limits, special criteria	
Certification required	
Standard Operating Procedure available for method	
One time project or on-going	
Special reporting parameters	
SAC created in LIMS	
Spike lists created in LIMS	
RL/MDL data entered into LIMS	

QA Review / Acceptance _____ **Date** _____

Figure 20-3.
Work Flow



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 North Canton follows a well-defined 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 Analyst and Group Leader 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 summarizes the schedule for routine maintenance. It is the responsibility of each Group Leader to ensure that instrument maintenance logs are kept for all equipment in his/her department. Preventative maintenance procedures may 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.)

21.2.4 Instrument maintenance logs are controlled and are used to document instrument problems, instrument repair and maintenance activities. Maintenance logs shall be kept for all major pieces of equipment. Instrument Maintenance Logbooks may also be 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, 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 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 Serial and Model Number)
- The date the instrument/equipment was put into use.
- If available, the condition when the instrument was received, e.g. new, used, reconditioned

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 shall 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 shall examine the effect of this defect on previous analyses (refer to Sections 12 and 13).

21.2.7 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. Back up instruments, which have been approved, for the analysis shall 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 shall be subcontracted using the procedures outlined in Section 8.

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, 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.

21.3.1 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 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.

21.3.2 pH, Conductivity, and Turbidity Meters

The pH meters used in the laboratory are accurate to ± 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.

21.3.3 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 has increments of 0.2 °C, and has a range applicable to all 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.

21.3.4 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 $4 \pm 2^{\circ}\text{C}$.

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.

21.3.5 Autopipettors, Dilutors, and Syringes

Mechanical volumetric dispensing devices including burettes (except Class A Glassware) are checked for accuracy at least quarterly. Glass micro-syringes are considered the same as Class A glassware.

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.

21.3.6 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.

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 will be reported with appropriate data qualifiers (refer to Section 13).

Note: Instruments are calibrated initially and as needed after that and at least annually.

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 NIST whenever possible. Dilution standards are prepared from stock standards purchased from commercial suppliers. A standard log is maintained for 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 will then involve 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.

21.4.2 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 multippeak 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 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 (e.g. EPA 547) 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 be 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 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).
- 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 will vary. The internal standard solution will contain one or more internal standards and the concentration of the individual internal standards may differ within the spiking solution. 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. The volume of the solution spiked into sample extracts should be such that minimal dilution of the extract occurs, e.g., 10 μ L 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 one 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 should produce 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 should result in a minimum response factor of approximately 0.01 for the least responsive target compound. Refer to SOP CA-Q-S-005, Calibration Curves, for specific calculations.

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 level 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 should be re-run immediately 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 will apply 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, 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. The changes to the upper end of the calibration range will affect the need to dilute samples above the range, while changes to the lower end will 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 0.990 or better using the least squares method to be considered acceptable.

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 **may 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 **may 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.

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 available electronically on the public drive.

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 Section 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 [state what your lab uses] 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. See specific methods in *Standard Methods for the Examination of Water and Wastewater* (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 Sample results within the established linear range do not need to be qualified.

21.4.3.3.3 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.

- 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, 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 every 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: ± 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 shall 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 shall be reanalyzed after a new calibration curve has been established, evaluated and accepted. Alternatively, a reporting limit standard may be analyzed to demonstrate that the laboratory can still support non-detects at their reporting limit.

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:

$$\% \text{ Drift} = \frac{\text{Result} - \text{True Value}}{\text{True Value}} \times 100$$

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 Section 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.

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).

21.5 POLICY ON 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 will 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. See the following SOPs for guidelines for making tentative identifications (CORP-MS-0001NC, GC/MS Analysis Based on Method 8270C, and CORP-MS-0002NC, Determination of Volatile Organics by GC/MS based on Method 8260B & 8260A).

21.6 POLICY ON 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 21.6.4 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 two tries) without clear documentation is not allowed. Necessary maintenance is performed and documented in the 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. 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 should be built into the instrument software.

21.6.5 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.

Example - Laboratory Equipment and Instrumentation

Instrument Type	Manufacturer	Model	Auto-sampler
Metals ICP	Thermo Jarrell Ash	Trace Analyzer 61E	Yes
	Thermo Jarrell Ash	Trace Analyzer 61E	Yes
Metals ICP/MS	Perkin Elmer (I-7)	ELAN 6100	Yes
	Thermo (I-8)	Series 2	Yes
Metals Mercury Analyzer	Leeman (CVAA)	PS200 II	Yes
	Leeman (CVAF) Low Level	Hydra AF Gold+, Model #112-00067-1	Yes
	Leeman (CVAA)	Hydra AA	Yes
GC/MS Semivolatiles	Agilent A4AG2	5975-C	NA
	Hewlett-Packard HP7	5973-6890	NA
	Hewlett-Packard HP8	5973-6890	NA
	Hewlett-Packard HP9	5973-6890	NA
	Hewlett-Packard HP10	5973-6890	NA
GC/MS Volatiles	Hewlett-Packard (UX2)	5971A-5890	Yes
	Hewlett-Packard (HP6)	5973-6890	Yes
	Hewlett-Packard (UX7)	5973-6890	Yes
	Hewlett-Packard (UX8)	5973-6890	Yes
	Hewlett-Packard (UX9)	5973-6890	Yes
	Hewlett-Packard (UX10)	5973-6890	Yes
	Agilent (UX11) (former HP)	5973-6890	Yes
	Agilent (UX12)	5973-6890	Yes
	Agilent (UX14)	5973-6890	Yes
	Agilent (UX15)	5973-6890	Yes
	Agilent (UX16)	5975-6890	Yes
GC/MS Volatiles Autosampler	OI Analytical (UX2)	4552	
	OI Analytical (HP6)	4552	
	OI Analytical (UX7)	4552	
	OI Analytical (UX8)	4552	
	OI Analytical (UX9)	4552	
	OI Analytical (UX10)	4552	
	OI Analytical (UX11)	4552	
	OI Analytical (UX12)	4552	
	OI Analytical (UX14)	4552	
	OI Analytical (UX15)	4552	
	OI Analytical (UX16)	4552	

Instrument Type	Manufacturer	Model	Auto-sampler
GC/MS Volatiles Purge and Trap	OI Analytical (UX2)	4560	
	OI Analytical (HP6)	4560	
	OI Analytical (UX7)	4660	
	OI Analytical (UX8)	4560	
	OI Analytical (UX9)	4560	
	OI Analytical (UX10)	4560	
	OI Analytical (UX11)	4560	
	OI Analytical (UX12)	4560	
	OI Analytical (UX14)	4560	
	OI Analytical (UX15)	4660	
	OI Analytical (UX16)	4660	
	EST (spare)	Encon	
WC Ion Chromatograph	Dionex	DX-320	NA
	Dionex	DX-120	NA
WC TOC	OI Analytical	1010 TOC Analyzer	
WC TOX	Thermo Electron	1200-S/N 973515	
	Thermo Electron	1200-S/N 2001.0174	
	Thermo Electron	1200-S/N 2005.0234	
WC UV/VIS	Milton Roy	Spectronic 401	No
	Genesys	Spectronic 20	No
WC Autotitrator	Man-Tech	PC – Titrate	
WC pH Meter	Orion pH Meter	250A	
	Orion (Ammonia ISE)	520A	
WC Dissolved Oxygen Meter	YSI	52C E	No
WC Turbidimeter	HF Scientific	Micro 100	No
WC BOD	Labtronics, Inc.	BOD	
WC Block Digester	Andrews	2210 Phenol	
	Andrews	2205 Ammonia	
	Lachat	BD46 TKN	

Instrument Type	Manufacturer	Model	Auto-sampler
WC Cyanide	Midi Serial #1000-99-01	PRG-2520-BL	
WC Conductivity	Man-Tech	4310	
WC Flashpoint	Petrolab (199443)	Petrotest	
	Herzog	HFP 339	
WC Discrete Analyzer	Kone	Konelab 200	
	Kone	Konelab 250	
WC Residual Chlorine Meter	Hanna	HI 93701	
	Hanna	HI 93701	
WC TRAACS	Bran & Luebbe	800	
Ext. 6-Position Accelerated Soxhlet Extractor	Gerhardt Soxtherm	6 units	
Extractions Sonicators	Misonix	3000	NA
	Fisher	550	NA
GC Semivolatiles	Hewlett-Packard (P1)	6890 EPC & Dual ECD Y-Splitter	Yes
	Hewlett-Packard (P2)	6890 EPC & Dual ECD Y-Splitter	Yes
	Hewlett-Packard (P3)	6890 EPC & Dual ECD Y-Splitter	Yes
	Hewlett-Packard (P4)	6890 EPC & Dual ECD Y-Splitter	Yes
	Hewlett-Packard (P5)	6890 EPC & Dual ECD Y-Splitter	Yes
	Hewlett-Packard (P6)	6890 EPC & Dual FID	Yes
	Hewlett-Packard (P7)	6890 EPC & Dual FID	Yes
	Hewlett-Packard (P9)	6890 EPC & Dual ECD Y-Splitter	Yes
	Hewlett-Packard (P10)	6890 EPC & Dual ECD Y-Splitter	Yes

Instrument Type	Manufacturer	Model	Auto-sampler
GC Semivolatiles (Cont'd)	Hewlett-Packard (P11)	6890 EPC & Dual ECD Y-Splitter	Yes
GC Semivolatiles HPLC	Hewlett-Packard (L2)	HPLC 1100	Yes
	Hewlett-Packard (L3)	HPLC 1100	Yes
GC Volatiles	Agilent (A)	6890 PID/FID	Yes
	Hewlett-Packard (O)	6890 Dual PID/Hall	Yes
	Hewlett-Packard (P)	6890 PID/HALL	Yes
	Hewlett-Packard (Y)	6890N PID/FID	Yes
	Hewlett-Packard (P8)	6890 EPC & Dual FID	Yes
GC Volatiles Auto Sampler	OI Analytical (O)	Archon	
	OI Analytical (Y)	4552	
	Varian (A)	Archon	
	Varian (P)	4552	
GC Volatiles Purge & Trap	Tekmar (O)	4560	
	Tekmar (P)	3000	
	OI Analytical (A)	4560	
	OI Analytical (Y)	3000	
GM Bedford	Agilent	6890N	Yes
	Agilent	6890N	Yes
	Agilent	6890N	Yes
	Misonix Sonicator	3000	NA

Table 21-2.

Example – Laboratory Balance Inventory

Instrument	Location	Manufacturer	Model Number	Type	Serial Number
B001	MS Volatiles	OHAUS	E400D	Top Loader	3317
B002	MS Volatiles	Mettler	AC 100	Analytical	B01177
B005	Metals	Sartorius	R300S	Analytical	38020045
B006	Wet Chemistry	American Scientific	SP180	Analytical	2904794
B007	High Hazard Room	American Scientific	ER-180A	Analytical	2904257
B008	Waste Building (no log book)	Howe Richardson	XL 5401		A029390
B009	Leachates (Extractions)	OHAUS	GT 4800	Top Loader	1687
B010	Wet Chemistry	Mettler	PJ 3600	Top Loader	G29475
B011	Extractions	American Scientific	SP 180	Analytical	2902127
B013	Extractions	OHAUS	TS 4KD	Top Loader	1936
B018	GC Volatiles	American Scientific	DTL 350	Analytical	10594
B019	GC Volatiles	Sartorius	L2200S	Top Loader	36020158
B022	Wet Chemistry	Mettler	PM300	Top Loader	F03040
B023	Wet Chemistry	OHAUS	TS 400S	Top Loader	3608
B024	Wet Chem Prep Room	Mettler	AE 100	Analytical	C25750
B025	Extractions	Mettler	PM 4600	Top Loader	G64548
B027	Wet Chemistry	American Scientific	SP 180	Analytical	2904154
B028	Wet Chemistry	Denver Instrument	M Series	Analytical	P119656
B030	Metals (new 9/5/6)	AND	EK410i	Top Loader	P1841870
B031	Metals (new 9/5/6)	AND	EK410i	Top Loader	P1841872
B032	Metals	OHAUS	ARC120	Top Loader	H28312
B033	Leachates (Extractions)	OHAUS	ARC120	Top Loader	H27612

Table 21-3.

Example – Laboratory Refrigerators, Freezers, Ovens, Thermometers, Steambaths, Hotblocks, and Hotplates

Instrument Type	Manufacturer / Model	Location	Tracking No.
Refrigerators	American Scientific CLP	BNA	R015
	VWR Scientific	BNA	R027
	Frigidaire	BNA	R031
	GE GPC	Extractions	R007
	Hotpoint “D”	Extractions	R008
	GE	Extractions	R010
	Hotpoint CLP Pest.	GC Semi	R016
	VWR	GC Semi	R021
	Frigidaire	GC Semi	R030
	Kelvinator	GC Semi	R032
	BB	GC VOA	R017
	True	MS Voa	R028
	Cryo Frig A CLP	MS Voa	R001
	Mr. Winter	MS Voa	R002
	#203 Enseco	MS Voa	R003
	Hotpoint “OO”	Org Haz	R012
	Kelvinator	Org Prep	R011
	Frigidaire	R&D	R013
	Sample Walk-In 2 nd	Sample Receiving	R018
	LaCross	Sample Receiving	R019
	Walk-In CLP	Sample Receiving	R020
	True	Sample Receiving	R025
	Baxter Cryo	Sample Receiving	R033
	True	Wet Chem	R024
	Frigidaire BOD	Wet Chem	R023
	Baxter Cryo Fridge BOD	Wet Chem	R029
	Frigidaire BOD	Wet Chem	R034
	Fisher Scientific 307 BOD	Wet Chem	R004
Ovens	TDS	Wet Chem	O-001
	WWR	Wet Chem	O-002
	TSS	Wet Chem	O-003
	TDS Evap	Wet Chem	O-004
	Lindberg/Blue	Wet Chem	O-006

Instrument Type	Manufacturer / Model	Location	Tracking No.
Freezers	Kenmore	BNA	F005
	Frigidaire	BNA	F012
	Hotpoint "D"	Extractions	F007
	Magic Chef	GC Semi	F004
	Marvel HPLC	GC Semi	F013
	GE	GC VO	F002
	Kenmore Side/Side	MS Voa	F001
	Frigidaire	MS Voa	F011
	Frigidaire	MS Voa	F016
	Kelvinator	Org Prep	F003
	Crosley	Receiving - Warehouse	F008
	GE	Receiving - Warehouse	F009
	Frigidaire	Receiving - Warehouse	F014
NIST Thermometers	Ertco S/N 69887 Product 15041D		92564
	Ertco S/N NB179010 Product N/A		167513
	Ertco S/N 3053 Product ASTM 068C-BF		15169121
	Ertco S/N 3243 Product ASTM 62C		163757
Steambaths		Extractions	A
		Extractions	B
		Extractions	C
Hotblocks		Metals	0
		Metals	1
		Metals	2
		Metals	3
		Metals	4
		Metals	A1
		Metals	A2
		Metals	B1
		Metals	C1
		Metals	C2
		Metals	C3
		Metals	C4
Hotplate		Metals	1-A
		Metals TCLP	9 K

Table 21-4.

Example: 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	Lubricate pump	
Check tubing		

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	Manufacturer cleaning and calibration
Clean pan and weighing compartment	

**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 21-5.

Example: Periodic Equipment Calibrations

Type of Equipment	Calibration Requirements
Balances	• Must be serviced and calibrated annually by an approved vendor. • Calibration must be checked daily or before use by analyst with weight(s) classified as Class 1 (formerly termed Class S) by NIST or Class 1 traceable. Acceptance criteria vary according to weight used and accuracy of balance. Acceptance criteria must be documented in the log. • All Class 1 weights must be certified by an outside vendor every three years. • All non-Class 1 weights must be checked annually against NIST Class 1 weights annually.
Thermometers	• Working glass thermometers must be calibrated against a certified. • NIST thermometer at least annually as described in operation-specific SOPs. • Working non-glass thermometers must be calibrated against a certified NIST thermometer annually as described in operation-specific SOPs. • The NIST thermometer must be recertified every three years.
Refrigerators/ Freezers	• Thermometers must be immersed in a liquid such as mineral oil or glycol. • Temperature of units used for sample or standard storage must be checked daily as described in operation-specific SOPs. Refrigerator acceptance limits: $4^{\circ}\text{C} \pm 2^{\circ}\text{C}$. Freezer acceptance limits: $< -10^{\circ}\text{C}$
Ovens	• Temperature of units must be checked daily or before use. • Acceptance limits vary according to use as described in operation-specific SOPs, and must be documented in the temperature log.
Micropipettes	• Calibrations are checked gravimetrically as required by the operation-specific SOP. • Must be calibrated at the frequency (normally quarterly) required by the manufacturer at a minimum.
Syringes, Volumetric Glassware and Graduated Glassware	• All syringes and volumetric glassware are purchased as Class A items. • Class A items are certified by the manufacturer to be within $\pm 1\%$ of the measured volume; therefore, calibration of these items by TestAmerica laboratories is not required. • All analysts are trained in the proper use and maintenance of measuring devices to ensure the measurement of standards, reagents, and sample volumes are within method tolerances.

Table 21-6.

Example: 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, " $r^3 \geq 0.995$ "	9056A	5 levels plus a blank, " $r^3 \geq 0.995$ "
	Continuing	300.0A	Level every 10 samples $\pm 10\%$ 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 " $r^3 \geq 0.995$ "	--	N/A
	Continuing	410.4 SM5220D	One level every 10 samples $\pm 10\%$ of true value	--	N/A
	Ending	410.4 SM5220D	One level $\pm 10\%$ 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 $"r" \geq 0.995$	9056A 9251	<u>Method 9056:</u> 3 levels plus a blank <u>Method 9252:</u> 5 levels plus blank $"r" \geq 0.995$
	Continuing	300.0A 325.2	One level every 10 samples $\pm 10\%$ of true value	9056A 9251	<u>Method 9056:</u> One per batch of 20 samples, $\pm 10\%$ of true value <u>Method 9252:</u> One level every 10 samples, $\pm 10\%$ of true value
	Ending	300.0A 325.2	One level every 10 samples $\pm 10\%$ of true value	9056A 9251	<u>Method 9056 :</u> N/A <u>Method 9252:</u> One level $\pm 10\%$ of true value
Chromium Cr ⁺⁶	Initial	3500 Cr-D	3 levels plus blank	7196A	5 levels plus blank $"r" \geq 0.995$
	Continuing	3500 Cr-D	One level every 10 samples $\pm 10\%$ of true value	7196A	One level every 10 samples $\pm 15\%$
	Ending	3500 Cr-D	One level $\pm 10\%$ of true value	7196A	One level $\pm 15\%$
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	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	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	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	<u>Method 300.0A:</u> 5 levels plus blank "r" ³ ≥ 0.995 <u>Method 375.4:</u> 3 levels plus blank "r" ³ ≥ 0.995	9038 9056A	<u>Method 9038:</u> 3 levels plus a blank for every hour of continuous sample analysis. <u>Method 9056:</u> 3 levels plus a blank

Analysis	Calibration	NPDES ¹		RCRA (SW846) ²	
		Method	Requirement	Method	Requirement
Sulfate (Cont'd)	Continuing	300.0A 375.4	<u>Method 300.0A:</u> One mid-level after every 10 samples ± 10% of true value <u>Method 375.4:</u> One level every 3 or 4 samples ± 10% of true value	9038 9056A	<u>Method 9038:</u> Independent-prepared check standard every 15 samples <u>Method 9056:</u> 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	<u>Method 376.1:</u> 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	<u>Method 376.1:</u> N/A	9030B/ 9034 9030A	This is a colorimetric titration. Therefore, calibration is not applicable.
	Ending	376.1	<u>Method 376.1:</u> 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	6010B	One level and blank. ICV RSD <5% from replicate
	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 $\pm 10\%$ of true value "r" ³ ≥ 0.995	7470A 7471A	5 levels plus blank ICV $\pm 10\%$ of true value "r" ³ ≥ 0.995
	Continuing	245.1* 1631E	Daily or every 10 samples, whichever is more frequent $\pm 20\%$ of true value	7470A 7471A	Every 10 samples $\pm 20\%$ of true value
	Ending	245.1 1631E	$\pm 20\%$ of true value	7470A 7471A	$\pm 20\%$ of original prepared standard
	Other	245.1 1631E	<u>Annually:</u> MDL	7470A 7471A	<u>Annually:</u> MDL

* 245.1 continuing – Initial CCV $\pm 5\%$ 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 CORP-GC-0001)
	Continuing	608	One or more calibration standards analyzed daily. % D $\pm$ 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 $\pm$ 10% at 2 mg and $\pm$ 0.5% at 1000 mg or recalibrate balance	9071B	Calibrate analytical balance at 2 mg and 1000 mg Calibration must be $\pm$ 10% at 2 mg and $\pm$ 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 $\leq$ 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 $\leq$ 35%, use avg RF. Otherwise, calibration curve employed.	8260B	Minimum of 5 levels, %RSD for RF for CCCs ⁴ < 30.0% SPCCs ⁵ : RF $\geq$ 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 $\geq$ 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 22

MEASUREMENT TRACEABILITY (NELAC 5.5.6)

22.1 OVERVIEW

Traceability of measurements shall 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 shall 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. 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. The following definitions are provided by the American Association for Laboratory Accreditation (A2LA):

“Traceability is the property of a measurement result whereby it can be related to stated references, usually national or international standards, through an unbroken chain of comparisons, each step in the chain having stated uncertainties.” There are six essential elements:

- An unbroken chain of comparison
- A calculated measurement uncertainty for each step in the chain to allow for an overall uncertainty calculation
- Documentation of each step in each calibration report
- All steps in the chain are performed by individuals with evidence of technical competence and accredited by a recognized accreditation body
- Reference to International Standard (SI) units
- Recalibration at appropriate intervals to preserve traceability

Calibration is defined as “determining and documenting the deviation of the indication of a measuring instrument (or the stated value of a material measure) from the conventional ‘true’ value of the measurand.”

Uncertainty is defined as “a parameter associated with the result of a measurement that characterizes the dispersion of the value that could reasonably be attributed to the measurand.” Measurement of Uncertainty is discussed in Section 20 of this QA Manual.

22.2 NIST-TRACEABLE WEIGHTS AND THERMOMETERS

Reference standards of measurement shall 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. Refer to Section 21 for calibration of weights and thermometers.

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.

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, 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 Table 9-1 in Section 9 for general storage requirements and Table 22-1 for additional storage information. Please refer to SOP NC-QA-0017, Standards and Reagents, for additional details. 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 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 will 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 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.

22.4.2 All standards, reagents, and reference materials must be clearly labeled with a minimum of the following information:

- Expiration date
- Standard ID (from the Standards Logbook)
- Special health/safety warnings if applicable

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)
- 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
- 3) According to Table 22-1

SECTION 23.0

SAMPLING (NELAC 5.5.7)

23.1 OVERVIEW

TestAmerica North Canton provides sampling services. Sampling procedures are described in SOP NC-SC-0006, Sample Procurement Protocol.

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 available from the vendor electronically, or stored 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

Container orders are prepared using the laboratory Shipping Department, SOP NC-QA-0012. 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 will be analyzed along with the field samples (exception soil samples where the blank is aqueous).

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 should 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 will also be affected by the site and sample handling conditions evaluated by the other types of blanks. The sampling time for the equipment blank should begin when the equipment is rinsed and the water is collected. Equipment blank collection for low level mercury is generally done at a clean facility rather than at the sample collection site.

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 should be 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 will be used for sampling. 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 holding time compliance based on the date and specific time of analysis compared to the time of sampling regardless of how long the holding time is.

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.

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 (refer to Tables 23-1 to 23-7) or preservation requirements are not met, the reports will 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.

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. In that regard the following guidelines apply to analysts:

Analysts must 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.

23.6.1 For water samples, before taking each aliquot for analysis, invert the sample container end-over-end three times and immediately pour off the aliquot. Especially when suspended solids are present, adequate mixing of the sample is extremely important.

23.6.2 For solid samples, when volatile organics are not requested, if the solid can be mixed, stir before removing the aliquot. When possible stir the entire sample in the sample container. Remove small increments of sample from different parts of the container to make up the complete subsample.

- For soil samples, avoid debris in the subsample aliquot as much as possible (e.g. gravel, sticks, roots and grass); note this information in the sample preparation record.

23.6.3 For solid samples, when volatile organics analysis is requested, the sample should be manipulated as little as possible. In most cases, the sample will arrive already preserved or in an EnCore™ sampler of the correct mass (requiring quick preservation of the entire amount). If the client requests volatiles on a solid sample which has been collected in a jar and is in a common container from which aliquots for other test methods must be taken, login should deliver the container to the volatiles department for preparing a proper aliquot prior to any other aliquots being taken out.

23.6.4 For multiphasic samples, the client should instruct the laboratory as to the intent of the testing and how to handle the sample. If the entire sample is to be accounted for, and the phases do not mix easily with inversion/stirring, such that a representative aliquot can be taken, the analyst must record the percent by volume of each phase. The analysis must be conducted on each phase separately; the final results are combined mathematically, weighting the individual phase results by volume. One exception to this procedure is the situation addressed in the TCLP and SPLP methods for wastes containing free liquids. However, if the leachate and final filtrate are not miscible, it is necessary to combine mathematically the concentrations of the two (or more) solutions by volume. A laboratory coliwasa can be used to subsample a multilayer liquid sample when appropriate.

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 23-1.

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°C H ₂ SO 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 ₂ SO 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	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 ₂ SO 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 ₂ SO ₄ to pH < 2, 28 days	9065	1 liter glass recommended, Cool to 4°C, H ₂ SO 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 ₂ SO 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 ₂ SO 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 ₂ SO ₄ to pH < 2, 28 days	9060 Walkley-Black	100 mL glass or 40 mL VOA vials, Cool to 4°C, H ₂ SO 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 ₂ SO 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 ≤ 2, 6 months	6010B 6020	1 liter glass or polyethylene container, HNO ₃ to pH ≤ 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	245.1 245.7 1631E	250 mL glass or polyethylene container, HNO ₃ to pH ≤ 2, 28 days	7470A	1 liter glass or polyethylene container, HNO ₃ to pH ≤ 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.
- 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 23-2.

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 ₂ SO ₄ to pH <2 28 days	9071B	1 liter glass, Cool, 0-4°C HCl or H ₂ SO 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 ≤ 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.)

Table 23-3.

Sample Containers, Preservatives, and Holding Times for TCLP¹ and SPLP²

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 24

HANDLING OF SAMPLES (NELAC 5.5.8)

Sample management procedures at TestAmerica North Canton 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 can be 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 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 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 COC is stored with project information and the report.

24.1.2 Legal / Evidentiary Chain-of-Custody

The lab does not accept samples that require legal chain-of-custody.

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 shall 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-0005, Sample Receiving and Sample Control, describes the laboratory's sample receipt procedure.

24.2.1 Laboratory Receipt

Samples shall 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 shall 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 shall be stored in the project file. An example CRF is shown in Figure 24-4. The report narrative will include an explanation of sample receiving related anomalies.

24.2.2 Exceptions or Discrepancies

TestAmerica reserves the right to reject samples for any of the following reasons:

- No custody seals as required by project
- No chain of custody documentation provided
- Preservation inappropriate for analysis requested
- Sample container inappropriate for analysis requested
- Sample received out of holding time for analysis requested
- Incomplete sample information provided
- Discrepancies between COC and sample labels
- Samples have high levels of polychlorinated dibenzo-p-dioxins/ dibenzo furans (PCDD/PCDFs)
- Samples have a high level gross alpha or beta radiation
- Samples are from a site known to contain chemical warfare agents (CWAs) and the samples have not been screened for them.
- Samples containing high levels of PCBs, Cyanides, Sulfides, and Hydrofluoric Acid.
- Tissue samples that may contain viruses harmful to humans.
- Samples with percent levels of Target analytes must be discussed in advance before receipt.

These or any other project exceptions or discrepancies are discussed with the client and agreed upon action taken.

24.2.3 Sample Log-In

Sample log-in activities at TestAmerica North Canton are fully documented in SOP NC-SC-0005, Sample Receiving and Sample Control. The following is a general description of the log-in process:

- 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
- 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)

24.3 SAMPLE ACCEPTANCE POLICY

The laboratory has a written sample acceptance policy outlined in SOP NC-SC-0005, Sample Receiving and Sample Control, that clearly outlines the circumstances under which samples shall 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 will 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.

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 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.

The primary considerations for sample storage are:

- Maintenance at the method prescribed temperature, if required
- Maintenance of sample integrity through adequate protection from contamination from outside sources or from cross-contamination of samples. Low-level and high-level samples,

when known, must be stored separately. Samples and standards must be stored in separate refrigerators or freezers. Storage areas for volatile organic test requests must be monitored weekly by the analysis of a holding (refrigerator) blank (an aliquot of contaminant-free water stored in a VOA vial)

- Security of samples within the laboratory.

The requirements listed in Tables 23-1 through 23-3 for temperatures and holding times shall be used. Placing of samples in the proper storage environment is the responsibility of sample control personnel. TestAmerica will assign individuals the responsibility of notifying the Group/Team Leaders or their designees if there are any samples which must be analyzed immediately because of holding time requirements.

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

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.

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). 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.

24.7 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-0005, Sample Receiving and Sample Control, and the Facility Addendum to the Corporate

Safety Manual.) 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.

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 24-1. Example: Chain of Custody (COC)

[illegible]

Figure 24-2.

Example: Custody Seals

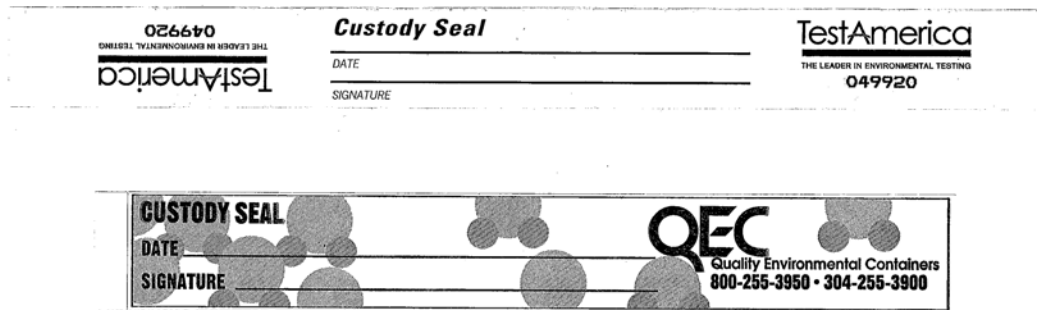


Figure 24-3.

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 24-4.

Example: Cooler Receipt Form (Page 1)

TestAmerica Cooler Receipt Form/Narrative North Canton Facility		Lot Number: _____	
Client _____		Project _____ Quote # _____	
Cooler Received on _____		Opened on _____ By _____	
FedEx ☐ Client Drop Off ☐ UPS ☐		DHL ☐ FAS ☐ TestAmerica Courier ☐	
Stetson ☐ US Cargo ☐		Other _____ (Signature)	
TestAmerica Cooler # _____		Foam Box ☐ Client Cooler ☐ Other _____	
1. Were custody seals on the outside of the cooler? Yes ☐ No ☐		Intact? Yes ☐ No ☐ NA ☐	
If YES, Quantity _____			
Were custody seals on the outside of cooler signed and dated?		Yes ☐ No ☐ NA ☐	
Were custody seals on the bottles?		Yes ☐ No ☐	
If YES, are there any exceptions _____			
2. Shipper's packing slip attached to this form?		Yes ☐ No ☐	
3. Did custody papers accompany the sample(s)? Yes ☐ No ☐		Relinquished by client? Yes ☐ No ☐	
4. Did you sign 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 and/or tags be reconciled with the COC?		Yes ☐ No ☐	
9. Were samples at the correct pH upon receipt?		Yes ☐ No ☐ NA ☐	
10. Were correct bottles used for the tests 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? Yes ☐ No ☐		Were VOAs on the COC? Yes ☐ No ☐	
Contacted PM _____ Date _____ by _____ via Voice Mail ☐ Verbal ☐ 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.			
16. SAMPLE PRESERVATION			
Sample(s) _____ were further preserved in sample receiving to meet recommended pH level(s). Nitric Acid Lot #071707-HNO ₃ - Sulfuric Acid Lot # 092006-H ₂ SO ₄ ; Sodium Hydroxide Lot # 122805 -NaOH; Hydrochloric Acid Lot # 092006-HCl; Sodium Hydroxide and Zinc Acetate Lot # 050205-CH ₃ COO ₂ ZN/NaOH			
What time was preservative added to sample(s)? _____			
Sample(s) _____ were received with bubble > 6 mm in diameter (Notify PM)			
Client ID	pH	Date	Initials

SECTION 25.0

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 may 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 one for each batch of samples--not to exceed 20 environmental samples.
- 25.3.1.4** Evaluation criteria and corrective action for method blanks is defined in the specific standard operating procedure for each analysis. Generally, corrective action is taken if the concentration of a target analyte in the blank is at or above the reporting limit as established by the method or regulation. Refer to Policy QA-003, TestAmerica North Canton Quality Control Program.

- The source of contamination is investigated
- Measures are taken to minimize or eliminate the source of the contamination
- Affected samples are reprocessed or the results are qualified on the final report.

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.

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. 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. Trip Blanks are also sometimes referred to as Travel Blanks.

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 SOP NC-QA-0020, Laboratory Holding Blanks, for additional information on holding blank criteria.

25.3.8 **Field blanks**, equipment blank and trip blanks, when received, are analyzed in the same manner as other field samples. When known, blanks 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".

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
- 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.

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, 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.
- 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** 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.).
- 25.4.1.5** 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 samples; not to exceed 20 environmental samples.
- 25.4.1.6** 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.

25.4.1.6.1 For methods that have 1-10 target analytes, spike all components.

25.4.1.6.2 For methods that include 11-20 target analytes, spike at least 10 or 80%, whichever is greater.

25.4.1.6.3 For methods with more than 20 target analytes, spike at least 16 components.

25.4.1.6.4 Exception: Due to analyte incompatibility in pesticides, Toxaphene and Chlordane are only spiked at client request based on specific project needs.

25.4.1.6.5 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.

25.4.1.7 **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 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 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 of the listed

components (see LCS analytes in Section 25.4.1.6 above) 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.

- 25.5.1.4** The percent recovery calculation for matrix spikes is essentially the same as the calculation shown in Section 25.4.1.7, 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 shall be reported, with data qualifiers, to the client whose sample produced poor recovery.

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 In most organic analyses, 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). The acceptance criteria in most methods are 50% to 200% of the responses in the mid-point of the corresponding calibration curve. Consult the method-specific SOPs for details on the internal standard compounds, calculations and acceptance criteria.

25.5.4.2 When the internal standard recoveries fall outside these limits, if there are not obvious chromatographic interferences, reanalyze the sample to confirm a possible matrix effect. If the recoveries confirm or there was obvious interference, results are reported from the original analysis and a qualifier is added. If the reanalysis meets internal standard recovery criteria, the second run is reported (or both are reported if requested by the client).

25.6 ACCEPTANCE CRITERIA (CONTROL LIMITS)

25.6.1 Each individual analyte in the LCS, MS, or Surrogate Spike are evaluated against the control limits as published in the test method. Where there are no established acceptance criteria, the laboratory calculates control limits with the use of control charts or, in some cases, utilizes client project specific or regulatory mandated 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.

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 should 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 should the results all be from a similar matrix, but the spiking levels should also be approximately the same (within a factor of 2). Similarly, the matrix spike and surrogate results should 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 should try and avoid 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, will lead to unrealistically narrow criteria. For a 99% confidence interval, one 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 shall be discarded if they were the result of human or mechanical error or sample concentration exceeded spike level by $> 4x$.

25.6.3 Laboratory generated % Recovery acceptance (control) limits are generally established by taking ± 3 Standard Deviations (99% confidence level) from the average recovery of a minimum of 20-30 data points (more points are preferred).

25.6.3.1 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).

25.6.3.2 In-house limits cannot be any wider than those mandated in a regulated analytical method.

25.6.3.3 The lowest acceptable recovery limit will be 10% (the analyte must be detectable).

25.6.3.4 The maximum acceptable recovery limit will be 200%.

25.6.3.5 The maximum acceptable RPD limit will be 30% for organic methods and 20% for inorganic methods. The minimum RPD limit is 10%.

25.6.3.6 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.

25.6.4 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-0018, Statistical Evaluation of Data and Development of Control Charts, for details.

25.6.4.1 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.

25.6.5 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 should be 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:

25.6.5.1 The analyte results are below the reporting limit and the LCS is above the upper control limit.

25.6.5.2 If the analytical results are above the relevant regulatory limit and the LCS is below the lower control limit.

25.6.5.3 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

25.6.5.3.1 Marginal exceedances are recovery exceedances between 3 SD and 4 SD from the mean recovery limit (NELAC).

25.6.5.3.2 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.

25.6.5.3.3 Though marginal exceedances may be allowed, the data must still be qualified to indicate it is outside of the normal limits.

25.6.6 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 reanalyzed to prove matrix interference. A more detailed discussion of acceptance criteria and corrective action can be found in Appendix 4 and in Section 13.

25.6.7 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. If the reanalysis meets surrogate recovery criteria, the second run is reported (or both are reported if requested by the client).

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.

25.8 ADDITIONAL PROCEDURES TO ASSURE QUALITY CONTROL

25.8.1 The laboratory has written 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 Sections 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 laboratory sample acceptance policy is included in Section 24.

SECTION 26.0

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 a conflict between the client requested formats and accreditation requirements or data usability information, accreditation requirements and data usability information will take precedence over client requests. 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 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, reviewed, and signed by the appropriate Project Manager. At a minimum, the standard laboratory report shall contain the following information:

26.2.1 A report title with a "Sample Result" header.

26.2.2 Each report page printed 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 at the bottom of each page. The report is sequentially paginated. The final page of the report is labeled as "End of Report".

26.2.4 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).

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.

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. This may be accomplished in a narrative or by attaching sample login sheets (refer to Section 26.2.4 – Item 3, regarding additional addenda).

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 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.

26.2.19 When NELAC accreditation is required, the lab shall certify that the test results meet all requirements of NELAC or provide reasons and/or justification if they do not.

26.2.20 The laboratory includes a cover page.

26.2.21 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.

26.2.22 When Soil samples are analyzed, a specific identification as to whether soils are reported on a “wet weight” or “dry weight” basis.

26.2.23 Appropriate laboratory certification number for the state of origin of the sample, if applicable.

26.2.24 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 will follow once all of the work has been completed.

26.2.25 Any out-of-network subcontracted analysis results are provided as a separate report on the official letterhead of the subcontractor. All in-network subcontracting is clearly identified on the report as to which laboratory performed a specific analysis.

26.3 REPORTING LEVEL OR REPORT TYPE

TestAmerica North Canton 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 26.2
- 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 26.7.

26.3.1 Electronic Data Deliverables (EDDs)

EDDs are routinely offered as part of TestAmerica’s services. TestAmerica North Canton offers a variety of EDD 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 shall 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.

26.4 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. Refer to Appendix 8 for a list of the laboratory's standard footnotes and qualifiers.

26.4.1 Numeric results with values outside of the calibration range, either high or low are qualified as 'estimated'.

26.4.2 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.

26.4.3 Where applicable, a statement on the estimated uncertainty of measurements; information on uncertainty is needed when a client's instructions so require.

26.4.4 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 will 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.

26.5 ENVIRONMENTAL TESTING OBTAINED FROM SUBCONTRACTORS

If TestAmerica North Canton is not able to provide the client the requested analysis, the samples would be 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.

26.6 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.

26.6.1 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."

26.7 **FORMAT OF REPORTS**

The format of reports are designed to accommodate each type of environmental test carried out and to minimize the possibility of misunderstanding or misuse.

26.8 **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 13).

If, after issuance of a report, TestAmerica North Canton observes any mistake that affects the results reported or the QC interpretation of those results, the client will be notified. After issuance of the report, the laboratory report remains unchanged. Any material amendments to a report after issue made only in the form of a further document, or data transfer must include the statement "Supplement to Test Report" or otherwise identified.

When the report is re-issued, a notation 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 re-issue.

26.9 **POLICIES ON CLIENT REQUESTS FOR AMENDMENTS**

26.9.1 **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 will reanalyze samples at a client's request with the following caveats. Client specific arrangements for reanalysis protocols can be established.

- 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 $\leq 5\times$ the reporting limit, the original analysis will be 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 will investigate the discrepancy and reanalyze 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 will typically be 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 Group Leader, QA Manager, or Laboratory Director/Manager if unsure.

26.9.2 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.

26.9.3 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 QA.

Appendix 1. TestAmerica Ethics Policy CA-L-P-001

**TESTAMERICA
ETHICS POLICY No. CA-L-P-001**

Refer to CA-L-P-001 for complete policy.

**TestAmerica
EMPLOYEE ETHICS STATEMENT**

I understand that TestAmerica is committed to ensuring the highest standard of quality and integrity of the data and services provided to our clients. I have read the Ethics Policy of the Company.

- With regard to the duties I perform and the data I report in connection with my employment at the Company, I agree that:*
- I will not intentionally report data values that are inconsistent with the actual values observed or measured.*
- I will not intentionally report the dates, times, sample or QC identifications, or method citations of data analyses that are not the actual dates, times, sample or QC identifications, or method citations.*
- I will not intentionally misrepresent another individual's work as my own or represent my own work as someone else's.*
- I will not intentionally misrepresent any data where data does not meet Method or QC requirements. If it is to be reported, I will report it with all appropriate notes and/or qualifiers; I shall not modify data (either sample or QC data) unless the modification can be technically justified through a measurable analytical process, such as one deemed acceptable to the laboratory's Standard Operating Procedures, Quality Assurance Manual or Technical Director. All such modifications must be clearly and thoroughly documented in the appropriate laboratory notebooks/worksheets and/or raw data and include my initials or signature and date.*
- I shall not make false statements to, or seek to otherwise deceive, members of Management or their representatives, agents, or clients/customers. I will not, through acts of commission, omission, erasure, or destruction, improperly report measurement standards, quality control data, test results or conclusions.*
- I shall not compare or disclose results for any Performance Testing (PT) sample, or other similar QA or QC requirements, with any employee of any other laboratory, including any other TestAmerica laboratory, prior to the required submission date of the results to the person, organization, or entity supplying the PT sample.*
- I shall immediately inform my supervisor or other member of management regarding any intentional or unintentional reporting of my own inauthentic data. Such report shall be given both orally and in writing to the supervisor or other member of management contacted and to the local Quality Assurance Manager. The Quality Assurance Manager will initial and date the information and return a copy to me. I shall not condone any accidental or intentional reporting of inauthentic data by other employees and will immediately report its occurrence. If I have actual knowledge of such acts committed by any other employees, and I do not report such information to designated members of Management, it shall be considered as serious as if I personally committed the offense. Accordingly, in that event, I understand that I may be subject to immediate termination of employment.*

- *I understand that if any supervisor, manager, or representative of TestAmerica management instructs, requests, or directs me to perform any of the aforementioned improper laboratory practices, or if I am in doubt or uncertain as to whether or not such laboratory practices are proper, I will not comply. In fact, I must report such event to all appropriate members of Management including, but not limited to, the Lab Director, all supervisors and managers with direct line reporting relationship between me and the Lab Director, and the local Quality Assurance representative, excluding such individuals who participated in such perceived improper instruction, request, or directive. In addition, I may contact Corporate Quality Assurance / Ethics Compliance Officer(s) for assistance.*
- *I understand the critical importance of accurately reporting data, measurements, and results, whether initially requested by a client, or retained by TestAmerica and submitted to a client at a later date, or retained by TestAmerica for subsequent internal use;*
- *I will not share the pricing or cost data of Vendors or Suppliers with anyone outside of the TestAmerica family of companies.*
- *I shall not accept gifts of a value that would adversely influence judgment.*
- *I shall avoid conflicts of interest and report any potential conflicts to the management (e.g. employment or consulting with competitors, clients, or vendors).*
- *I shall not participate in unfair competition practices (e.g. slandering competitors, collusion with other labs to restrict others from bidding on projects).*
- *I shall not misrepresent certifications and status of certifications to clients or regulators.*
- *I shall not intentionally discharge wastes illegally down the drain or onto the ground.*
- *I understand that any attempt by management or an employee to circumvent these policies will be subject to disciplinary action.*

As a TestAmerica employee, I understand that I have the responsibility to conduct myself with integrity in accordance with the ethical standards described in the Ethics Policy. I will also report any information relating to possible kickbacks or violations of the Procurement Integrity Act, or other questionable conduct in the course of sales or purchasing activities. I will not knowingly participate in any such activity and will report any actual or suspected violation of this policy to management.

I understand that if my job includes supervisory responsibilities, I shall not instruct, request, or direct any subordinate to perform any laboratory practice which is unethical or improper. Also, I shall not discourage, intimidate, or inhibit an employee who may choose to appropriately appeal my supervisory instruction, request, or directive which the employee perceives to be improper, nor retaliate against those who do.

The Ethics Policy has been explained to me by my supervisor or at a training session, and I have had the opportunity to ask questions if I did not understand any part of it. I understand that any violation of this policy subjects me to disciplinary action, which can include termination of my employment. In addition, I understand that any violation of this policy which relates to work under a government contract or subcontract could also subject me to the potential for prosecution under federal law.

EMPLOYEE SIGNATURE _____

Date _____

Supervisor/Trainer: _____

Date _____

Work Instruction No. CA-WI-005

TestAmerica
CONFIDENTIALITY AND PROPRIETARY INFORMATION AGREEMENT

TestAmerica and their predecessors, in their businesses, have developed and use commercially valuable technical and non-technical information and to guard the legitimate interests of TestAmerica and its clients, it is necessary to protect certain information as confidential and proprietary.

I, _____, understand and acknowledge that during the term of my employment by TestAmerica, I will be privy to and entrusted with certain confidential information and trade secrets of TestAmerica and its clients.

Confidential information and trade secrets include, but are not limited to: customer and client lists; price lists; marketing and sales strategies and procedures; operational and equipment techniques; standard operating procedures; business plans and systems; quality control procedures and systems; special projects and technological research, including projects, research and reports for any government entity or client; client's plans and processes; client's manner of operation; the trade secrets of clients; client's data; vendor or supplier pricing; employee lists and personal information, and any other records, data, files, drawings, inventions, discoveries, applications, or processes which are not in the public domain.

I agree as follows:

1. I will not in any way, during the term of my employment, or at any time thereafter, except as authorized in writing by the Legal Department of TestAmerica or the client where client data is involved, disclose to others, use for my own benefit, remove from TestAmerica's premises (except to the extent off-site work is approved by my supervisor), copy or make notes of any confidential information and/or trade secrets of TestAmerica or its clients, excepting only that information which may be public knowledge. Technical and business information of any previous employer or other third party which I may disclose to TestAmerica shall be limited to that which was acquired legitimately and disclosed to me without restriction as to secrecy.
2. I agree that all inventions (whether or not patentable) conceived or made by me during the period of my employment by TestAmerica shall belong to TestAmerica, provided such inventions grow out of my work for TestAmerica and are related to the business of TestAmerica. I agree to disclose and assign such inventions to TestAmerica. In California, this provision shall not apply to any invention which qualifies fully under Section 2870 of the California Labor Code.
3. On termination of my employment from TestAmerica, I will deliver to TestAmerica all documents, records, notes, data, memoranda, files, manuals, equipment and things of any nature which relate in any way to confidential information and/or trade secrets of TestAmerica or its clients and which are in my possession or under my control.
4. I agree that during the period of my employment and for one (1) year from and after the termination (for any reason) of my employment with TestAmerica, I shall not directly or indirectly (without first obtaining the written permission of TestAmerica), recruit for employment, or induce to terminate his or her employment with TestAmerica, any person who is an active employee of TestAmerica on the last day of my employment with TestAmerica.
5. I acknowledge that if I were to breach any provision of this Confidentiality Agreement, money damages will be inadequate, and I hereby agree that TestAmerica shall be entitled, where appropriate, to specific performance and/or injunctive relief (i.e. to require me to comply with this Agreement). I further acknowledge that the willingness of TestAmerica to hire me or to continue my employment constitutes full and adequate consideration for the agreements, and obligations to which I have agreed as set forth in this document.

I have executed this Agreement, intending to be legally bound.

Printed Name

Signature

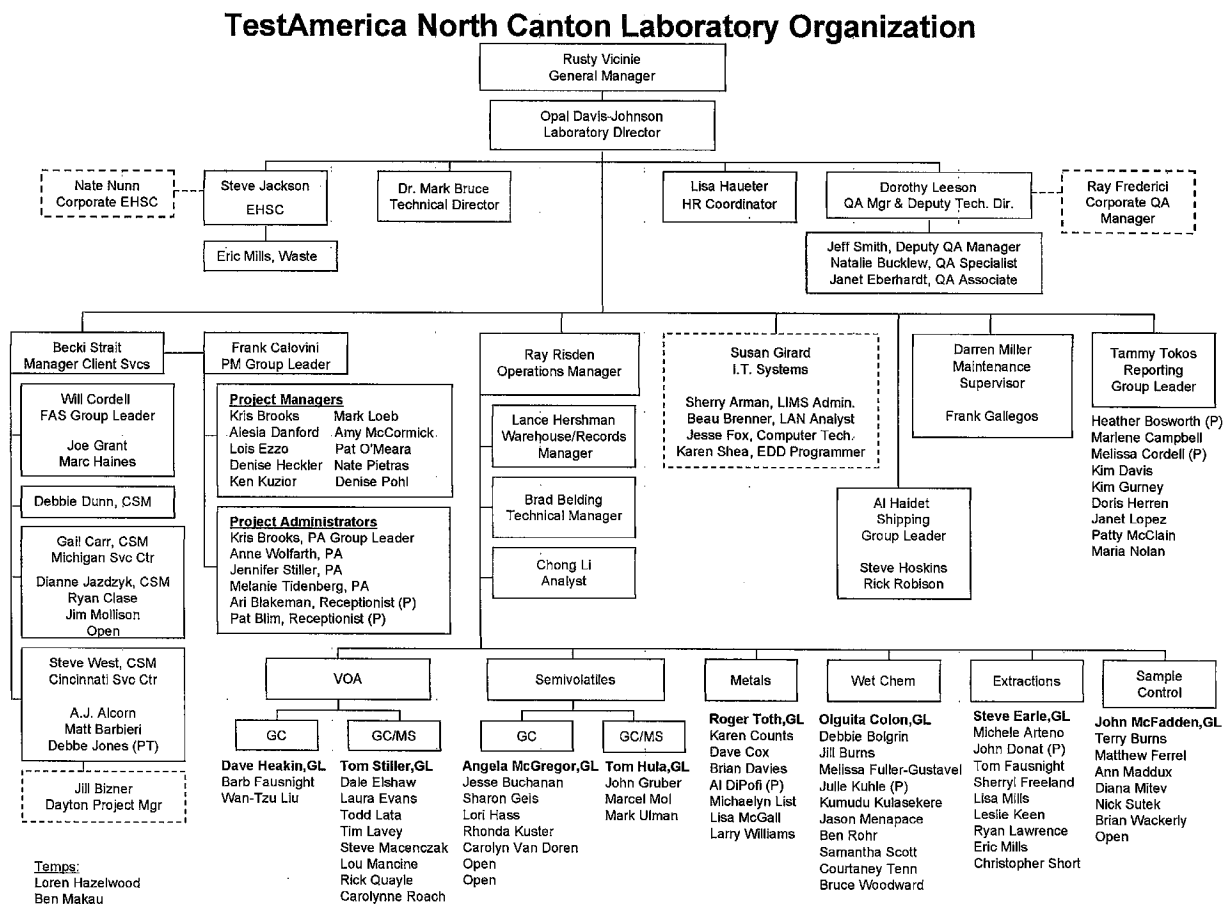
Date

Work Instruction No. CA-WI-006

Appendix 2.

Example Laboratory Organization Chart

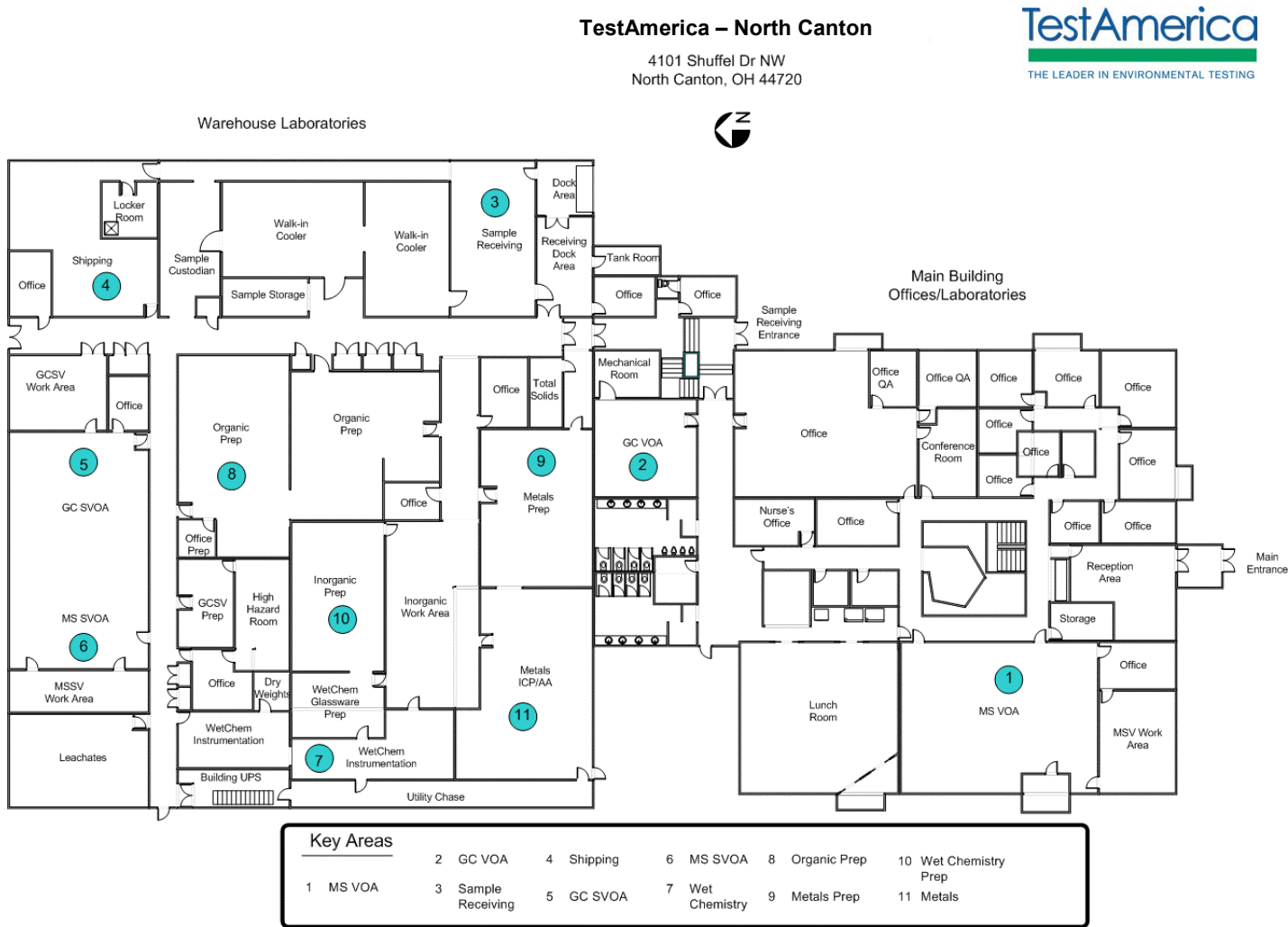
(The most current chart can be obtained from the QA Manager or Lab Director/Manager)



11/06/07

Appendix 3.

Laboratory Floor Plan



Appendix 4.

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	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 Fluorescence

Analytical Parameters	Matrix	Fields of Testing			
			CWA	RCRA (SW846)	Other
Mercury, Low Level (CVAFS)	Water		EPA 245.7	--	EPA 1631E

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	---
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	---

Analytical Parameters	Matrix	Fields of Testing			
			CWA	RCRA (SW846)	Other
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	---
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	---

Analytical Parameters	Matrix	Fields of Testing			
			CWA	RCRA (SW846)	Other
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	---
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	---

Analytical Parameters	Matrix	Fields of Testing			
			CWA	RCRA (SW846)	Other
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	---

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 245.7	---	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 Dissolved Gases RSK-175	Water		---	EPA 8015B (M)	WI DRO
	Waste		---	EPA 8015B (M)	---
	Water		---	---	SOP
Formaldehyde Carbonyl Compounds	Water		---	EPA 8315	---

Appendix 5.

Laboratory Reporting Limits

**General Chemistry
Reporting Limits (RL), Method Reference**

Test Method	Analyte	RL	Units
305.1/SM2310B	Acidity	5	mg/L
310.1/SM2320B	Alkalinity, Total	5	mg/L
7063	Arsenic Speciation	2	ug/L
300.0 9056A	Bromide Chloride Nitrate Fluoride Sulfate Ortho-Phosphate Nitrite	0.5 1 0.1 1 1 0.5 0.1	mg/L mg/L mg/L mg/L mg/L mg/L mg/L
405.1/SM5210B	Biochemical Oxygen Demand	2	mg/L
410.4/SM5220D	Chemical Oxygen Demand (COD)	10	mg/L
325.2/9251	Chloride, Automated	1	mg/L
330.5 SM4500CL-G	Chlorine, Total Residue	0.2	mg/L
9012A/335.2 335.4 SM4500CN-E	Cyanide, Total	0.01	mg/L
340.2 SM4500F-C, ISE	Fluoride	0.1	mg/L
130.2/SM2340B	Hardness, as CaCO ₃	5	mg/L
1664A	n-Hexane Extractable Material n-Hexane Extractable Material, SGT	5 10	mg/L mg/L
7196A	Hexavalent Chromium	0.02	mg/L
3500 Cr D	Hexavalent Chromium	0.02	mg/L
353.2 SM4500NO ₃ E	Nitrite Nitrate Nitrate/Nitrite	0.1 0.1 0.1	mg/L mg/L mg/L
350.2 SM4500NH ₃ -E,F	Nitrogen, as Ammonia	1	mg/L

General Chemistry
Reporting Limits (RL), Method Reference

Test Method	Analyte	RL	Units
9065	Phenols, Total	0.02	mg/L
9045B, C SM4500H-B	pH (solid)	--	No Units
4500	Phosphorus as Orthophosphate	0.1	mg/L
365.2 365.1	Ortho-Phosphate Phosphorus, as ortho-Phosphate ortho-Phosphate, Dissolved	0.1 0.1 0.1	mg/L mg/L mg/L
120.1 SM2510B	Specific Conductance	1	umhos/cm
9050A	Specific Conductance	1	umhos/cm
160.1 SM2540E	Solids (Residue), Dissolved Filterable Solids, Total Dissolved	10 10	mg/L mg/L
375.4	Sulfate	5	mg/L
9038	Sulfate	5	mg/L
376.1	Sulfide, Total	1	mg/L
9030A	Sulfide, Total	1	mg/L
9030A	Sulfide, Acid-Insoluble	1	mg/L
351.3	Total Kjeldahl Nitrogen	3	mg/L
415.1 SM5310D	Total Organic Carbon	1	mg/L
9060	Total Organic Carbon	1	mg/L
9020B	Total Organic Halogens	30	ug/L
180.1	Turbidity	0.5	NTU

Metals – ICP, CVAA 6000, 7000, 200 Series
Reporting Limits (RL), Method Reference^{1, 2}

	Analyte	RL	Units
Trace Water	Antimony	0.01	mg/L
	Arsenic	0.01	mg/L
	Cadmium	0.002	mg/L
	Chromium	0.005	mg/L
	Cobalt	0.007	mg/L
	Lead	0.003	mg/L
	Molybdenum	0.01	mg/L
	Selenium	0.005	mg/L
	Silver	0.005	mg/L
	Thallium	0.01	mg/L
	Vanadium	0.007	mg/L
Trace Solid	Antimony	1	mg/kg
	Arsenic	1	mg/kg
	Cadmium	0.2	mg/kg
	Chromium	0.5	mg/kg
	Cobalt	5	mg/kg
	Lead	0.3	mg/kg
	Molybdenum	1	mg/kg
	Selenium	0.5	mg/kg
	Silver	0.5	mg/kg
	Thallium	1	mg/kg
	Vanadium	5	mg/kg
Water	Aluminum	0.2	mg/L
	Antimony	0.06	mg/L
	Arsenic	0.3	mg/L
	Barium	0.2	mg/L
	Beryllium	0.005	mg/L
	Boron	0.2	mg/L
	Cadmium	0.005	mg/L
	Calcium	5	mg/L
	Chromium	0.01	mg/L
	Cobalt	0.05	mg/L
	Copper	0.025	mg/L
	Iron	0.1	mg/L
	Lead	0.1	mg/L
	Magnesium	5	mg/L
	Manganese	0.015	mg/L
	Molybdenum	0.04	mg/L
	Nickel	0.04	mg/L
	Potassium	5	mg/L
	Selenium	0.25	mg/L
	Silver	0.01	mg/L
	Sodium	5	mg/L
	Thallium	0.2	mg/L
	Tin	0.1	mg/L
	Titanium	0.05	mg/L
	Vanadium	0.05	Mg/L
	Zinc	0.02	mg/L

TABLE 8.2-10-2
Metals – ICP, CVAA 6000, 7000, 200 Series
Reporting Limits (RL), Method Reference ^{1,2}

	Analyte	RL	Units
Solid	Aluminum	20	mg/kg
	Antimony	6	mg/kg
	Arsenic	30	mg/kg
	Barium	20	mg/kg
	Beryllium	0.5	mg/kg
	Boron	20	mg/kg
	Cadmium	0.5	mg/kg
	Calcium	500	mg/kg
	Chromium	1	mg/kg
	Cobalt	5	mg/kg
	Copper	2.5	mg/kg
	Iron	10	mg/kg
	Lead	10	mg/kg
	Magnesium	500	mg/kg
	Manganese	1.5	mg/kg
	Molybdenum	4	mg/kg
	Nickel	4	mg/kg
	Potassium	500	mg/kg
	Selenium	25	mg/kg
	Silver	1	mg/kg
	Sodium	500	mg/kg
	Thallium	200	mg/kg
	Tin	10	mg/kg
	Titanium	5	mg/kg
	Vanadium	5	mg/kg
	Zinc	2	mg/kg
Water	Mercury	0.0002	mg/L
Solid	Mercury	0.1	mg/kg

Metals – ICPMS, 6020
Reporting Limits (RL), Method Reference ^{1,2}

	Analyte	RL	Units
Water	Aluminum	50	ug/L
	Antimony	2	ug/L
	Arsenic	5	ug/L
	Barium	1	ug/L
	Beryllium	1	ug/L
	Cadmium	1	ug/L
	Chromium	2	ug/L
	Cobalt	1	ug/L
	Copper	2	ug/L
	Iron	20	ug/L
	Lead	1	ug/L
	Manganese	1	ug/L
	Molybdenum*	10	ug/L
	Nickel	2	ug/L
	Selenium*	5	ug/L
	Silver	1	ug/L
	Thallium	1	ug/L
	Tin*	10	ug/L
	Vanadium*	5	ug/L
	Zinc	10	Ug/L

*Molybdenum, Selenium, Tin, and Vanadium are not on the 6020 method list

Solid	Antimony	0.2	mg/kg
	Arsenic	0.5	mg/kg
	Barium	0.1	mg/kg
	Beryllium	0.1	mg/kg
	Cadmium	0.1	mg/kg
	Chromium	0.2	mg/kg
	Cobalt	0.1	mg/kg
	Copper	0.2	mg/kg
	Lead	0.1	mg/kg
	Manganese	0.1	mg/kg
	Molybdenum*	0.2	mg/kg
	Nickel	0.1	mg/kg
	Selenium*	0.5	mg/kg
	Silver	0.1	mg/kg
	Thallium	0.1	mg/kg
	Tin*	1	mg/kg
	Vanadium*	0.5	mg/kg
	Zinc	1	mg/kg

*Molybdenum, Selenium, Tin, and Vanadium are not on the 6020 method list

Metals – ICPMS, 200.8
Reporting Limits (RL), Method Reference ^{1,2}

	Analyte	RL	Units
Water	Aluminum	50	ug/L
	Antimony	2	ug/L
	Arsenic	5	ug/L
	Barium	1	ug/L
	Beryllium	1	ug/L
	Cadmium	1	ug/L
	Chromium	2	ug/L
	Cobalt	1	ug/L
	Copper	2	ug/L
	Iron	20	ug/L
	Lead	1	ug/L
	Manganese	1	ug/L
	Molybdenum	10	ug/L
	Nickel	2	ug/L
	Selenium	2	ug/L
	Silver	1	ug/L
	Thallium	1	ug/L
	Tin*	10	ug/L
	Vanadium	5	ug/L
	Zinc	10	ug/L

*Tin is not on the 200.8 method list

Metals – Low-Level Mercury, 1631E & 245.7
Reporting Limits (RL), Method Reference ^{1,2}

	Analyte	RL	Units
1631E Water	Mercury	0.5	ng/L
245.7 Water	Mercury	5	ng/L

MS Semivolatiles – Method 8270C
Reporting Limits (RL), Method Reference ^{1,2}

	Analyte	RL	Units
Water	Acenaphthene	10	ug/L
	Acenaphthylene	10	ug/L
	Anthracene	10	ug/L
	Benzo(a)anthracene	10	ug/L
	Benzo(b)fluoranthene	10	ug/L
	Benzo(k)fluoranthene	10	ug/L
	Benzo(ghi)perylene	10	ug/L
	Benzo(a)pyrene	10	ug/L
	Bis(2-chloroEthoxy)methane	10	ug/L
	bis(2-Chloroethyl) ether	10	ug/L
	bis(2-Ethylhexyl) phthalate	10	ug/L
	4-Bromophenyl phenyl ether	10	ug/L
	Butyl benzyl phthalate	10	ug/L
	Carbazole	10	ug/L
	4-Chloroaniline	10	ug/L
	4-Chloro-3-methylphenol	10	ug/L
	2-Chloronaphthalene	10	ug/L
	2-Chlorophenol	10	ug/L
	4-Chlorophenyl phenyl ether	10	ug/L
	Chrysene	10	ug/L
	Dibenz(a,h)anthracene	10	ug/L
	Dibenzofuran	10	ug/L
	Di-n-butyl phthalate	10	ug/L
	1,2-Dichlorobenzene	10	ug/L
	1,3-Dichlorobenzene	10	ug/L
	1,4-Dichlorobenzene	10	ug/L
	3,3'-Dichlorobenzidine	50	ug/L
	2,4-Dichlorophenol	10	ug/L
	Diethyl phthalate	10	ug/L
	2,4-Dimethylphenol	10	ug/L
	Dimethyl phthalate	10	ug/L
	4,6-Dinitro-2-methylphenol	50	ug/L
	2,4-Dinitrophenol	50	ug/L
	2,4-Dinitrotoluene	10	ug/L
	2,6-Dinitrotoluene	10	ug/L
	Di-n-octyl phthalate	10	ug/L
	Fluoranthene	10	ug/L
	Fluorene	10	ug/L
	Hexachlorobenzene	10	ug/L
	Hexachlorobutadiene	10	ug/L
	Hexachloro-cyclopentadiene	50	ug/L
	Hexachloroethane	10	ug/L
	Indeno(1,2,3-cd)pyrene	10	ug/L
	Isophorone	10	ug/L
	2-Methylnaphthalene	10	ug/L
	2-Methylphenol	10	ug/L
	4-Methylphenol	10	ug/L
	Naphthalene	10	ug/L
	2-Nitroaniline	50	ug/L

	Analyte	RL	Units
	3-Nitroaniline	50	ug/L
	4-Nitroaniline	50	ug/L
	Nitrobenzene	10	ug/L
	2-Nitrophenol	10	ug/L
	4-Nitrophenol	50	ug/L
	N-Nitrosodiphenylamine	10	ug/L
	N-Nitrosodi-n-propylamine	10	ug/L
	Pentachlorophenol	10	ug/L
	Phenanthrene	10	ug/L
	Phenol	10	ug/L
	2,2'-Oxybis(1-Chloropropane)	10	ug/L
	Pyrene	10	ug/L
	1,2,4-Trichlorobenzene	10	ug/L
	2,4,5-Trichlorophenol	10	ug/L
	2,4,6-Trichlorophenol	10	ug/L
Water Surrogates	2-Fluorobiphenyl		
	2-Fluorophenol		
	2,4,6-Tribromophenol		
	Nitrobenzene-d5		
	Phenol-d5		
	Terphenyl-d14		

MS Semivolatiles – Method 8270C
Reporting Limits (RL), Method Reference ^{1,2}

	Analyte	RL	Units
Solid	Acenaphthene	330	ug/kg
	Acenaphthylene	330	ug/kg
	Anthracene	330	ug/kg
	Benzo(a)anthracene	330	ug/kg
	Benzo(b)fluoranthene	330	ug/kg
	Benzo(k)fluoranthene	330	ug/kg
	Benzo(ghi)perylene	330	ug/kg
	Benzo(a)pyrene	330	ug/kg
	bis(2-Chloroethoxy)-methane	330	ug/kg
	bis(2-Chloroethyl) ether	330	ug/kg
	bis(2-Ethylhexyl) phthalate	330	ug/kg
	4-Bromophenyl phenyl ether	330	ug/kg
	Butyl benzyl phthalate	330	ug/kg
	Carbazole	330	ug/kg
	4-Chloroaniline	330	ug/kg
	4-Chloro-3-methylphenol	330	ug/kg
	2-Chloronaphthalene	330	ug/kg
	2-Chlorophenol	330	ug/kg
	4-Chlorophenyl phenyl ether	330	ug/kg
	Chrysene	330	ug/kg
	Dibenz(a,h)anthracene	330	ug/kg
	Dibenzofuran	330	ug/kg
	Di-n-butyl phthalate	330	ug/kg
	1,2-Dichlorobenzene	330	ug/kg
	1,3-Dichlorobenzene	330	ug/kg
	1,4-Dichlorobenzene	330	ug/kg
	3,3'-Dichlorobenzidine	1600	ug/kg
	2,4-Dichlorophenol	330	ug/kg
	Diethyl phthalate	330	ug/kg
	2,4-Dimethylphenol	330	ug/kg
	Dimethyl phthalate	330	ug/kg
	4,6-Dinitro-2-methylphenol	1600	ug/kg
	2,4-Dinitrophenol	1600	ug/kg
	2,4-Dinitrotoluene	330	ug/kg
	2,6-Dinitrotoluene	330	ug/kg
	Di-n-octyl phthalate	330	ug/kg
	Fluoranthene	330	ug/kg
	Fluorene	330	ug/kg
	Hexachlorobenzene	330	ug/kg
	Hexachlorobutadiene	330	ug/kg
	Hexachloro-cyclopentadiene	1600	ug/kg
	Hexachloroethane	330	ug/kg
	Indeno(1,2,3-cd)pyrene	330	ug/kg
	Isophorone	330	ug/kg
	2-Methylnaphthalene	330	ug/kg
	2-Methylphenol	330	ug/kg
	4-Methylphenol	330	ug/kg
	Naphthalene	330	ug/kg
	2-Nitroaniline	1600	ug/kg
	3-Nitroaniline	1600	ug/kg

	Analyte	RL	Units
	4-Nitroaniline	1600	ug/kg
	Nitrobenzene	330	ug/kg
	2-Nitrophenol	330	ug/kg
	4-Nitrophenol	1600	ug/kg
	N-Nitrosodiphenylamine	330	ug/kg
	N-Nitrosodi-n-propylamine	330	ug/kg
	Pentachlorophenol	330	ug/kg
	Phenanthrene	330	ug/kg
	Phenol	330	ug/kg
	2,2'-Oxybis(1-Chloropropane)	330	ug/kg
	Pyrene	330	ug/kg
	1,2,4-Trichlorobenzene	330	ug/kg
	2,4,5-Trichlorophenol	330	ug/kg
	2,4,6-Trichlorophenol	330	ug/kg
Solid Surrogates	2-Fluorobiphenyl		
	2-Fluorophenol		
	2,4,6-Tribromophenol		
	Nitrobenzene-d5		
	Phenol-d5		
	Terphenyl-d14		

MS Semivolatiles – Method 625
Reporting Limits (RL), Method Reference ^{1,2}

	Analyte	RL	Units
Water	Acenaphthene	10	ug/L
	Acenaphthylene	10	ug/L
	Anthracene	10	ug/L
	Benzo(a)anthracene	10	ug/L
	Benzo(b)fluoranthene	10	ug/L
	Benzo(k)fluoranthene	10	ug/L
	Benzo(ghi)perylene	10	ug/L
	Benzo(a)pyrene	10	ug/L
	bis(2-Chloroethoxy)-methane	10	ug/L
	bis(2-Chloroethyl) ether	10	ug/L
	bis(2-Chloroisopropyl) ether	10	ug/L
	bis(2-Ethylhexyl) phthalate	10	ug/L
	4-Bromophenyl phenyl ether	10	ug/L
	Butyl benzyl phthalate	10	ug/L
	4-Chloro-3-methylphenol	10	ug/L
	2-Chloronaphthalene	10	ug/L
	2-Chlorophenol	10	ug/L
	4-Chlorophenyl phenyl ether	10	ug/L
	Chrysene	10	ug/L
	Dibenz(a,h)anthracene	10	ug/L
	Di-n-butyl phthalate	10	ug/L
	1,2-Dichlorobenzene	10	ug/L
	1,3-Dichlorobenzene	10	ug/L
	1,4-Dichlorobenzene	10	ug/L
	3,3'-Dichlorobenzidine	10	ug/L
	2,4-Dichlorophenol	10	ug/L
	Diethyl phthalate	10	ug/L
	2,4-Dimethylphenol	10	ug/L
	Dimethyl phthalate	10	ug/L
	2-Methyl-4,6-dinitrophenol	50	ug/L
	2,4-Dinitrophenol	50	ug/L
	2,4-Dinitrotoluene	10	ug/L
	2,6-Dinitrotoluene	10	ug/L
	Di-n-octyl phthalate	10	ug/L
	Fluoranthene	10	ug/L
	Fluorene	10	ug/L
	Hexachlorobenzene	10	ug/L
	Hexachlorobutadiene	10	ug/L
	Hexachloroethane	10	ug/L
	Indeno(1,2,3-cd)pyrene	10	ug/L
	Isophorone	10	ug/L
	Naphthalene	10	ug/L
	Nitrobenzene	10	ug/L
	2-Nitrophenol	10	ug/L
	4-Nitrophenol	50	ug/L
	N-Nitrosodi-n-propylamine	10	ug/L
	Pentachlorophenol	10	ug/L
	Phenanthrene	10	ug/L
	Phenol	10	ug/L
	Pyrene	10	ug/L

Analyte	RL	Units
1,2,4-Trichlorobenzene	10	ug/L
2,4,6-Trichlorophenol	10	ug/L

**Water
Surrogates**

2-Fluorobiphenyl
2-Fluorophenol
2,4,6-Tribromophenol
Nitrobenzene-d5
Phenol-d5
Terphenyl-d14

MS Volatiles – Method 8260B
Reporting Limits (RL), Method Reference ^{1,2}

	Analyte	RL	Units
Water 25 mL	Benzene	1	ug/L
	Bromobenzene	1	ug/L
	Bromochloromethane	1	ug/L
	Bromodichloromethane	1	ug/L
	Bromoform	1	ug/L
	Bromomethane	1	ug/L
	n-Butylbenzene	1	ug/L
	sec-Butylbenzene	1	ug/L
	tert-Butylbenzene	1	ug/L
	Carbon tetrachloride	1	ug/L
	Chlorobenzene	1	ug/L
	Chlorodibromomethane	1	ug/L
	Chloroethane	1	ug/L
	Chloroform	1	ug/L
	Chloromethane	1	ug/L
	2-Chlorotoluene	1	ug/L
	4-Chlorotoluene	1	ug/L
	1,2-Dibromo-3-chloropropane	2	ug/L
	1,2-Dibromoethane	1	ug/L
	Dibromomethane	1	ug/L
	1,2-Dichlorobenzene	1	ug/L
	1,3-Dichlorobenzene	1	ug/L
	1,4-Dichlorobenzene	1	ug/L
	Dichlorodifluoro-methane	1	ug/L
	1,1-Dichloroethane	1	ug/L
	1,2-Dichloroethane	1	ug/L
	cis-1,2-Dichloroethene	1	ug/L
	trans-1,2-Dichloroethene	1	ug/L
	1,1-Dichloroethene	1	ug/L
	1,2-Dichloropropane	1	ug/L
	1,3-Dichloropropane	1	ug/L
	2,2-Dichloropropane	1	ug/L
	1,1-Dichloropropene	1	ug/L
	Ethylbenzene	1	ug/L
	Hexachlorobutadiene	1	ug/L
	Isopropylbenzene	1	ug/L
	p-Isopropyltoluene	1	ug/L
	Methylene chloride	1	ug/L
	Naphthalene	1	ug/L
	n-Propylbenzene	1	ug/L
	Styrene	1	ug/L
	1,1,1,2-Tetrachloroethane	1	ug/L
	1,1,2,2-Tetrachloroethane	1	ug/L
	Tetrachloroethene	1	ug/L
	Toluene	1	ug/L
	1,2,3-Trichlorobenzene	1	ug/L
	1,2,4-Trichlorobenzene	1	ug/L
	1,1,1-Trichloroethane	1	ug/L
	1,1,2-Trichloroethane	1	ug/L

Analyte	RL	Units
Trichloroethene	1	ug/L
Trichlorofluoromethane	1	ug/L
1,2,3-Trichloropropane	1	ug/L
1,2,4-Trimethylbenzene	1	ug/L
1,3,5-Trimethylbenzene	1	ug/L
Vinyl chloride	1	ug/L
m-Xylene & p-Xylene	1	ug/L
o-Xylene	1	ug/L

Water
25 mL
Surrogates

4-Bromofluorobenzene
1,2-Dichloroethane-d4
Toluene-d8
Dibromofluoromethane

MS Volatiles – Method 8260B
Reporting Limits (RL), Method Reference ^{1,2}

	Analyte	RL	Units
Water 5 mL	Benzene	5	ug/L
	Bromobenzene	5	ug/L
	Bromochloromethane	5	ug/L
	Bromodichloromethane	5	ug/L
	Bromoform	5	ug/L
	Bromomethane	5	ug/L
	n-Butylbenzene	5	ug/L
	sec-Butylbenzene	5	ug/L
	tert-Butylbenzene	5	ug/L
	Carbon tetrachloride	5	ug/L
	Chlorobenzene	5	ug/L
	Chlorodibromomethane	5	ug/L
	Chloroethane	5	ug/L
	Chloroform	5	ug/L
	Chloromethane	5	ug/L
	2-Chlorotoluene	5	ug/L
	4-Chlorotoluene	5	ug/L
	1,2-Dibromo-3-chloropropane	10	ug/L
	1,2-Dibromoethane	5	ug/L
	Dibromomethane	5	ug/L
	1,2-Dichlorobenzene	5	ug/L
	1,3-Dichlorobenzene	5	ug/L
	1,4-Dichlorobenzene	5	ug/L
	Dichlorodifluoro-methane	5	ug/L
	1,1-Dichloroethane	5	ug/L
	1,2-Dichloroethane	5	ug/L
	cis-1,2-Dichloroethene	5	ug/L
	trans-1,2-Dichloroethene	5	ug/L
	1,1-Dichloroethene	5	ug/L
	1,2-Dichloropropane	5	ug/L
	1,3-Dichloropropane	5	ug/L
	2,2-Dichloropropane	5	ug/L
	1,1-Dichloropropene	5	ug/L
	Ethylbenzene	5	ug/L
	Hexachlorobutadiene	5	ug/L
	Isopropylbenzene	5	ug/L
	p-Isopropyltoluene	5	ug/L
	Methylene chloride	5	ug/L
	Naphthalene	5	ug/L
	n-Propylbenzene	5	ug/L
	Styrene	5	ug/L
	1,1,1,2-Tetrachloroethane	5	ug/L
	1,1,1,2,2-Tetrachloroethane	5	ug/L
	Tetrachloroethene	5	ug/L
	Toluene	5	ug/L
	1,2,3-Trichlorobenzene	5	ug/L
	1,2,4-Trichlorobenzene	5	ug/L
	1,1,1-Trichloroethane	5	ug/L
	1,1,2-Trichloroethane	5	ug/L
	Trichloroethene	5	ug/L

Analyte	RL	Units
Trichlorofluoromethane	5	ug/L
1,2,3-Trichloropropane	5	ug/L
1,2,4-Trimethylbenzene	5	ug/L
1,3,5-Trimethylbenzene	5	ug/L
Vinyl chloride	5	ug/L
m-Xylene & p-Xylene	10	ug/L
o-Xylene	5	ug/L

Surrogates

4-Bromofluorobenzene
1,2-Dichloroethane-d4
Toluene-d8
Dibromofluoromethane

MS Volatiles – Method 8260B
Reporting Limits (RL), Method Reference ^{1,2}

	Analyte	RL	Units
Encore	Benzene	250	ug/kg
	Bromobenzene	250	ug/kg
	Bromochloromethane	250	ug/kg
	Bromodichloromethane	250	ug/kg
	Bromoform	250	ug/kg
	Bromomethane	250	ug/kg
	n-Butylbenzene	250	ug/kg
	sec-Butylbenzene	250	ug/kg
	tert-Butylbenzene	250	ug/kg
	Carbon tetrachloride	250	ug/kg
	Chlorobenzene	250	ug/kg
	Chlorodibromomethane	250	ug/kg
	Chloroethane	250	ug/kg
	Chloroform	250	ug/kg
	Chloromethane	250	ug/kg
	2-Chlorotoluene	250	ug/kg
	4-Chlorotoluene	250	ug/kg
	1,2-Dibromo-3-chloropropane	500	ug/kg
	1,2-Dibromoethane	250	ug/kg
	Dibromomethane	250	ug/kg
	1,2-Dichlorobenzene	250	ug/kg
	1,3-Dichlorobenzene	250	ug/kg
	1,4-Dichlorobenzene	250	ug/kg
	Dichlorodifluoro-methane	250	ug/kg
	1,1-Dichloroethane	250	ug/kg
	1,2-Dichloroethane	250	ug/kg
	cis-1,2-Dichloroethene	250	ug/kg
	trans-1,2-Dichloroethene	250	ug/kg
	1,1-Dichloroethene	250	ug/kg
	1,2-Dichloropropane	250	ug/kg
	1,3-Dichloropropane	250	ug/kg
	2,2-Dichloropropane	250	ug/kg
	1,1-Dichloropropene	250	ug/kg
	Ethylbenzene	250	ug/kg
	Hexachlorobutadiene	250	ug/kg
	Isopropylbenzene	250	ug/kg
	p-Isopropyltoluene	250	ug/kg
	Methylene chloride	250	ug/kg
	Naphthalene	250	ug/kg
	n-Propylbenzene	250	ug/kg
	Styrene	250	ug/kg
	1,1,1,2-Tetrachloroethane	250	ug/kg
	1,1,2,2-Tetrachloroethane	250	ug/kg
	Tetrachloroethene	250	ug/kg
	Toluene	250	ug/kg
	1,2,3-Trichlorobenzene	250	ug/kg
	1,2,4-Trichlorobenzene	250	ug/kg
	1,1,1-Trichloroethane	250	ug/kg
	1,1,2-Trichloroethane	250	ug/kg

	Analyte	RL	Units
	Trichloroethene	250	ug/kg
	Trichlorofluoromethane	250	ug/kg
	1,2,3-Trichloropropane	250	ug/kg
	1,2,4-Trimethylbenzene	250	ug/kg
	1,3,5-Trimethylbenzene	250	ug/kg
	Vinyl chloride	250	ug/kg
	m-Xylene & p-Xylene	500	ug/kg
	o-Xylene	250	ug/kg

Surrogates	4-Bromofluorobenzene
	1,2-Dichloroethane-d4
	Toluene-d8
	Dibromofluoromethane

MS Volatiles – Method 8260B
Reporting Limits (RL), Method Reference ^{1,2}

	Analyte	RL	Units
Low Level Encore	Benzene	5	ug/kg
	Bromobenzene	5	ug/kg
	Bromochloromethane	5	ug/kg
	Bromodichloromethane	5	ug/kg
	Bromoform	5	ug/kg
	Bromomethane	5	ug/kg
	n-Butylbenzene	5	ug/kg
	sec-Butylbenzene	5	ug/kg
	tert-Butylbenzene	5	ug/kg
	Carbon tetrachloride	5	ug/kg
	Chlorobenzene	5	ug/kg
	Chlorodibromomethane	5	ug/kg
	Chloroethane	5	ug/kg
	Chloroform	5	ug/kg
	Chloromethane	5	ug/kg
	2-Chlorotoluene	5	ug/kg
	4-Chlorotoluene	5	ug/kg
	1,2-Dibromo-3-chloropropane	10	ug/kg
	1,2-Dibromoethane	5	ug/kg
	Dibromomethane	5	ug/kg
	1,2-Dichlorobenzene	5	ug/kg
	1,3-Dichlorobenzene	5	ug/kg
	1,4-Dichlorobenzene	5	ug/kg
	Dichlorodifluoro-methane	5	ug/kg
	1,1-Dichloroethane	5	ug/kg
	1,2-Dichloroethane	5	ug/kg
	cis-1,2-Dichloroethene	5	ug/kg
	trans-1,2-Dichloroethene	5	ug/kg
	1,1-Dichloroethene	5	ug/kg
	1,2-Dichloropropane	5	ug/kg
	1,3-Dichloropropane	5	ug/kg
	2,2-Dichloropropane	5	ug/kg
	1,1-Dichloropropene	5	ug/kg
	Ethylbenzene	5	ug/kg
	Hexachlorobutadiene	5	ug/kg
	Isopropylbenzene	5	ug/kg
	p-Isopropyltoluene	5	ug/kg
	Methylene chloride	5	ug/kg
	Naphthalene	5	ug/kg
	n-Propylbenzene	5	ug/kg
	Styrene	5	ug/kg
	1,1,1,2-Tetrachloroethane	5	ug/kg
	1,1,2,2-Tetrachloroethane	5	ug/kg
	Tetrachloroethene	5	ug/kg
	Toluene	5	ug/kg
	1,2,3-Trichlorobenzene	5	ug/kg
	1,2,4-Trichlorobenzene	5	ug/kg
	1,1,1-Trichloroethane	5	ug/kg
	1,1,2-Trichloroethane	5	ug/kg
	Trichloroethene	5	ug/kg

	Analyte	RL	Units
	Trichlorofluoromethane	5	ug/kg
	1,2,3-Trichloropropane	5	ug/kg
	1,2,4-Trimethylbenzene	5	ug/kg
	1,3,5-Trimethylbenzene	5	ug/kg
	Vinyl chloride	5	ug/kg
	m-Xylene & p-Xylene	10	ug/kg
	o-Xylene	5	ug/kg

Surrogates	4-Bromofluorobenzene
	1,2-Dichloroethane-d4
	Toluene-d8
	Dibromofluoromethane

MS Volatiles – Method 8260B
Reporting Limits (RL), Method Reference ^{1,2}

	Analyte	RL	Units
Frozen Encore	Benzene	5	ug/kg
	Bromobenzene	5	ug/kg
	Bromochloromethane	5	ug/kg
	Bromodichloromethane	5	ug/kg
	Bromoform	5	ug/kg
	Bromomethane	5	ug/kg
	n-Butylbenzene	5	ug/kg
	sec-Butylbenzene	5	ug/kg
	tert-Butylbenzene	5	ug/kg
	Carbon tetrachloride	5	ug/kg
	Chlorobenzene	5	ug/kg
	Chlorodibromomethane	5	ug/kg
	Chloroethane	5	ug/kg
	Chloroform	5	ug/kg
	Chloromethane	5	ug/kg
	2-Chlorotoluene	5	ug/kg
	4-Chlorotoluene	5	ug/kg
	1,2-Dibromo-3-chloropropane	10	ug/kg
	1,2-Dibromoethane	5	ug/kg
	Dibromomethane	5	ug/kg
	1,2-Dichlorobenzene	5	ug/kg
	1,3-Dichlorobenzene	5	ug/kg
	1,4-Dichlorobenzene	5	ug/kg
	Dichlorodifluoro-methane	5	ug/kg
	1,1-Dichloroethane	5	ug/kg
	1,2-Dichloroethane	5	ug/kg
	cis-1,2-Dichloroethene	5	ug/kg
	trans-1,2-Dichloroethene	5	ug/kg
	1,1-Dichloroethene	5	ug/kg
	1,2-Dichloropropane	5	ug/kg
	1,3-Dichloropropane	5	ug/kg
	2,2-Dichloropropane	5	ug/kg
	1,1-Dichloropropene	5	ug/kg
	Ethylbenzene	5	ug/kg
	Hexachlorobutadiene	5	ug/kg
	Isopropylbenzene	5	ug/kg
	p-Isopropyltoluene	5	ug/kg
	Methylene chloride	5	ug/kg
	Naphthalene	5	ug/kg
	n-Propylbenzene	5	ug/kg
	Styrene	5	ug/kg
	1,1,1,2-Tetrachloroethane	5	ug/kg
	1,1,2,2-Tetrachloroethane	5	ug/kg
	Tetrachloroethene	5	ug/kg
	Toluene	5	ug/kg
	1,2,3-Trichlorobenzene	5	ug/kg
	1,2,4-Trichlorobenzene	5	ug/kg
	1,1,1-Trichloroethane	5	ug/kg
	1,1,2-Trichloroethane	5	ug/kg
	Trichloroethene	5	ug/kg

	Analyte	RL	Units
	Trichlorofluoromethane	5	ug/kg
	1,2,3-Trichloropropane	5	ug/kg
	1,2,4-Trimethylbenzene	5	ug/kg
	1,3,5-Trimethylbenzene	5	ug/kg
	Vinyl chloride	5	ug/kg
	m-Xylene & p-Xylene	10	ug/kg
	o-Xylene	5	ug/kg

Surrogates	4-Bromofluorobenzene
	1,2-Dichloroethane-d4
	Toluene-d8
	Dibromofluoromethane

MS Volatiles – Method 624
Reporting Limits (RL), Method Reference ^{1,2}

	Analyte	RL	Units
Water	Benzene	5	ug/L
	Bromodichloro-methane	5	ug/L
	Bromoform	5	ug/L
	Bromomethane	55	ug/L
	Carbon tetrachloride	5	ug/L
	Chlorobenzene	5	ug/L
	Dibromochloro-methane	5	ug/L
	Chloroethane	5	ug/L
	2-Chloroethyl vinyl ether	--	ug/L
	Chloroform	5	ug/L
	Chloromethane	5	ug/L
	1,2-Dichlorobenzene	5	ug/L
	1,3-Dichlorobenzene	5	ug/L
	1,4-Dichlorobenzene	5	ug/L
	1,1-Dichloroethane	5	ug/L
	1,2-Dichloroethane	5	ug/L
	trans-1,2-Dichloroethene	5	ug/L
	1,1-Dichloroethene	5	ug/L
	1,2-Dichloropropane	5	ug/L
	cis-1,3-Dichloropropene	5	ug/L
	trans-1,3-Dichloropropene	5	ug/L
	Ethylbenzene	5	ug/L
	Methylene chloride	5	ug/L
	1,1,2,2-Tetrachloroethane	5	ug/L
	Tetrachloroethene	5	ug/L
	Toluene	5	ug/L
	1,1,1-Trichloroethane	5	ug/L
	1,1,2-Trichloroethane	5	ug/L
	Trichloroethene	5	ug/L
	Trichlorofluoro-methane	5	ug/L
	Vinyl chloride	5	ug/L
Surrogates	Bromofluorobenzene		
	1,2-Dichloro-ethane-d4		
	Toluene-d8		

GC Semivolatiles – Method 8081A
Reporting Limits (RL), Method Reference ^{1,2}

	Analyte	RL	Units
Water	Aldrin	0.05	ug/L
	alpha-BHC	0.05	ug/L
	beta-BHC	0.05	ug/L
	delta-BHC	0.05	ug/L
	gamma-BHC (Lindane)	0.05	ug/L
	alpha-Chlordane	0.05	ug/L
	gamma-Chlordane	0.05	ug/L
	4,4'-DDD	0.05	ug/L
	4,4'-DDE	0.05	ug/L
	4,4'-DDT	0.05	ug/L
	Dieldrin	0.05	ug/L
	Endosulfan I	0.05	ug/L
	Endosulfan II	0.05	ug/L
	Endosulfan sulfate	0.05	ug/L
	Endrin	0.05	ug/L
	Endrin aldehyde	0.05	ug/L
	Endrin ketone	0.05	ug/L
	Heptachlor	0.05	ug/L
	Heptachlor epoxide	0.05	ug/L
	Methoxychlor	0.1	ug/L
	Toxaphene	2	ug/L
Surrogates	Decachloro-biphenyl		
	Tetrachloro-m-xylene		

GC Semivolatiles – 8081A
Reporting Limits (RL), Method Reference ^{1,2}

	Analyte	RL	Units
Solid	Aldrin	1.7	ug/kg
	alpha-BHC	1.7	ug/kg
	beta-BHC	1.7	ug/kg
	delta-BHC	1.7	ug/kg
	gamma-BHC (Lindane)	1.7	ug/kg
	alpha-Chlordane	1.7	ug/kg
	gamma-Chlordane	1.7	ug/kg
	4,4'-DDD	1.7	ug/kg
	4,4'-DDE	1.7	ug/kg
	4,4'-DDT	1.7	ug/kg
	Dieldrin	1.7	ug/kg
	Endosulfan I	1.7	ug/kg
	Endosulfan II	1.7	ug/kg
	Endosulfan sulfate	1.7	ug/kg
	Endrin	1.7	ug/kg
	Endrin aldehyde	1.7	ug/kg
	Endrin ketone	1.7	ug/kg
	Heptachlor	1.7	ug/kg
	Heptachlor epoxide	1.7	ug/kg
	Methoxychlor	3.3	ug/kg
	Toxaphene	67	ug/kg
Surrogates	Decachloro-biphenyl		
	Tetrachloro-m-xylene		

GC Semivolatiles – Method 8082
Reporting Limits (RL), Method Reference ^{1,2}

	Analyte	RL	Units
Water	Aroclor 1016	1	ug/L
	Aroclor 1221	1	ug/L
	Aroclor 1232	1	ug/L
	Aroclor 1242	1	ug/L
	Aroclor 1248	1	ug/L
	Aroclor 1254	1	ug/L
	Aroclor 1260	1	ug/L
	Aroclor 1262	1	ug/L
	Aroclor 1268	1	ug/L
Surrogates	Decachlorobi-phenyl Tetrachloro-m-xylene		
Solid	Aroclor 1016	33	ug/kg
	Aroclor 1221	33	ug/kg
	Aroclor 1232	33	ug/kg
	Aroclor 1242	33	ug/kg
	Aroclor 1248	33	ug/kg
	Aroclor 1254	33	ug/kg
	Aroclor 1260	33	ug/kg
	Aroclor 1262	33	ug/kg
	Aroclor 1268	33	ug/kg
Surrogates	Decachloro-biphenyl Tetrachloro-m-xylene		

GC Semivolatiles – Method 608
Reporting Limits (RL), Method Reference ^{1,2}

	Analyte	RL	Units
Water	Aldrin	0.05	ug/L
	alpha-BHC	0.05	ug/L
	beta-BHC	0.05	ug/L
	delta-BHC	0.05	ug/L
	gamma-BHC (Lindane)	0.05	ug/L
	Chlordane (technical)	0.5	ug/L
	4,4'-DDD	0.05	ug/L
	4,4'-DDE	0.05	ug/L
	4,4'-DDT	0.05	ug/L
	Dieldrin	0.05	ug/L
	Endosulfan I	0.05	ug/L
	Endosulfan II	0.05	ug/L
	Endosulfan sulfate	0.05	ug/L
	Endrin	0.05	ug/L
	Endrin aldehyde	0.05	ug/L
	Heptachlor	0.05	ug/L
	Heptachlor epoxide	0.05	ug/L
	PCB-1016	1	ug/L
	PCB-1221	1	ug/L
	PCB-1232	1	ug/L
	PCB-1242	1	ug/L
	PCB-1248	1	ug/L
	PCB-1254	1	ug/L
	PCB-1260	1	ug/L
	Toxaphene	2	ug/L
Surrogates	Decachloro-biphenyl		
	Tetrachloro-m-xylene		

GC Semivolatiles – Method 8151A
Reporting Limits (RL), Method Reference ^{1,2}

	Analyte	RL	Units
Water	2,4-D	4	ug/L
	Dalapon	2	ug/L
	2,4-DB	4	ug/L
	Dicamba	2	ug/L
	Dichlorprop	4	ug/L
	Dinoseb	0.6	ug/L
	MCPA	400	ug/L
	MCPP	400	ug/L
	2,4,5-TP (Silvex)	1	ug/L
	2,4,5-T	1	ug/L
Surrogate	2,4-Dichlorophenylacetic acid		
Solid	2,4-D	80	ug/kg
	Dalapon	40	ug/kg
	2,4-DB	80	ug/kg
	Dicamba	40	ug/kg
	Dichlorprop	80	ug/kg
	Dinoseb	12	ug/kg
	MCPA	8000	ug/kg
	MCPP	8000	ug/kg
	2,4,5-TP (Silvex)	20	ug/kg
	2,4,5-T	20	ug/kg
Surrogate	2,4-Dichlorophenylacetic acid		

GC Volatiles - Method 8021B
Reporting Limits (RL), Method Reference ^{1,2}

		Analyte	RL	Units
Water 5 mL Purge		Benzene	1	ug/L
		Bromobenzene	1	ug/L
		Bromochloromethane	1	ug/L
		Bromodichloromethane	1	ug/L
		Bromoform	1	ug/L
		Bromomethane	1	ug/L
		n-Butylbenzene	1	ug/L
		sec-Butylbenzene	1	ug/L
		tert-Butylbenzene	1	ug/L
		Carbon tetrachloride	1	ug/L
		Chlorobenzene	1	ug/L
		Chlorodibromomethane	1	ug/L
		Chloroethane	1	ug/L
		Chloroform	1	ug/L
		Chloromethane	1	ug/L
		2-Chlorotoluene	1	ug/L
		4-Chlorotoluene	1	ug/L
		1,2-Dibromo-3-chloropropane (DBCP)	1	ug/L
		1,2-Dibromoethane (EDB)	1	ug/L
		Dibromomethane	1	ug/L
		1,2-Dichlorobenzene	1	ug/L
		1,3-Dichlorobenzene	1	ug/L
		1,4-Dichlorobenzene	1	ug/L
		Dichlorodifluoromethane	1	ug/L
		1,1-Dichloroethane	1	ug/L
		1,2-Dichloroethane	1	ug/L
		cis-1,2-Dichloroethene	1	ug/L
		trans-1,2-Dichloroethene	1	ug/L
		1,1-Dichloroethene	1	ug/L
		1,2-Dichloropropane	1	ug/L
		1,3-Dichloropropane	1	ug/L
		2,2-Dichloropropane	1	ug/L
		cis-1,3-Dichloropropene	1	ug/L
		trans-1,3-Dichloropropene	1	ug/L
		1,1-Dichloropropene	1	ug/L
		Ethylbenzene	1	ug/
		Hexachlorobutadiene	1	ug/L
		Isopropylbenzene	1	ug/L
		p-Isopropyltoluene	1	ug/L
		Methylene chloride	5	ug/L
		Naphthalene	1	ug/L
		n-Propylbenzene	1	ug/L
		Styrene	1	ug/L
		1,1,1,2-Tetrachloroethane	1	ug/L
		1,1,2,2-Tetrachloroethane	1	ug/L
		Tetrachloroethene	1	ug/L
		Toluene	1	ug/L
		1,2,3-Trichlorobenzene	1	ug/L
		1,2,4-Trichlorobenzene	1	ug/L

GC Volatiles - Method 8021B
Reporting Limits (RL), Method Reference ^{1,2}

Analyte		RL	Units
1,1,1-Trichloroethane		1	ug/L
1,1,2-Trichloroethane		1	ug/L
Trichloroethene		1	ug/L
Trichlorofluoromethane		1	ug/L
1,2,3-Trichloropropane		1	ug/L
1,2,4-Trimethylbenzene		1	ug/L
1,3,5-Trimethylbenzene		1	ug/L
Vinyl chloride		1	ug/L
m-Xylene & p-Xylene		2	ug/L
o-Xylene		1	ug/L
Xylenes (total)		3	ug/L
Surrogates	1,4-Dichlorobutane		
	Trifluorotoluene		

GC Volatiles - Method 8021B
Reporting Limits (RL), Method Reference ^{1,2}

		Analyte	RL	Units
Solid 5 g		Benzene	1	ug/kg
		Ethylbenzene	1	ug/kg
		Methyl tert-butyl ether (MTBE)	1	ug/kg
		Methyl tert-butyl ether	1	ug/kg
		Naphthalene	1	ug/kg
		Toluene	1	ug/kg
		1,2,4-Trimethylbenzene	1	ug/kg
		1,3,5-Trimethylbenzene	1	ug/kg
		m-Xylene & p-Xylene	2	ug/kg
		o-Xylene	1	ug/kg
		Xylenes (total)	3	ug/kg
Surrogates		Trifluorotoluene		

GC Volatiles – Method 8021B
Reporting Limits (RL), Method Reference ^{1,2}

	Analyte	RL	Units
Solid Encore, Methanol Prep	Benzene	50	ug/kg
	Ethylbenzene	50	ug/kg
	Methyl tert-butyl ether (MTBE)	50	ug/kg
	Naphthalene	250	ug/kg
	Toluene	50	ug/kg
	1,2,4-Trimethylbenzene	50	ug/kg
	1,3,5-Trimethylbenzene	50	ug/kg
	m-Xylene & p-Xylene	100	ug/kg
	o-Xylene	50	ug/kg
	Xylenes (total)	150	ug/kg
Surrogates	Trifluorotoluene		

GC Volatiles – Method 8021B
Reporting Limits (RL), Method Reference ^{1,2}

	Analyte	RL	Units
Frozen Encore	Benzene	1	ug/kg
	Ethylbenzene	1	ug/kg
	Methyl tert-butyl ether (MTBE)	1	ug/kg
	Methyl tert-butyl ether	1	ug/kg
	Naphthalene	1	ug/kg
	Toluene	1	ug/kg
	1,2,4-Trimethylbenzene	1	ug/kg
	1,3,5-Trimethylbenzene	1	ug/kg
	m-Xylene & p-Xylene	2	ug/kg
	o-Xylene	1	ug/kg
	Xylenes (total)	3	ug/kg

Surrogates	Trifluorotoluene

GC Volatiles – Method 601
Reporting Limits (RL), Method Reference ^{1,2}

	Analyte	RL	Units
Water	Bromodichloromethane	1	ug/L
	Bromoform	1	ug/L
	Bromomethane	1	ug/L
	Carbon tetrachloride	1	ug/L
	Chlorobenzene	1	ug/L
	Dibromochloromethane	1	ug/L
	Chloroethane	1	ug/L
	2-Chloroethyl vinyl ether	5	ug/L
	Chloroform	1	ug/L
	Chloromethane	1	ug/L
	1,2-Dichlorobenzene	1	ug/L
	1,3-Dichlorobenzene	1	ug/L
	1,4-Dichlorobenzene	1	ug/L
	Dichlorodifluoromethane	1	ug/L
	1,1-Dichloroethane	1	ug/L
	1,2-Dichloroethane	1	ug/L
	trans-1,2-Dichloroethene	1	ug/L
	1,1-Dichloroethene	1	ug/L
	1,2-Dichloropropane	1	ug/L
	cis-1,3-Dichloropropene	1	ug/L
	trans-1,3-Dichloropropene	1	ug/L
	Methylene chloride	5	ug/L
	1,1,2,2-Tetrachloroethane	1	ug/L
	Tetrachloroethene	1	ug/L
	1,1,1-Trichloroethane	1	ug/L
	1,1,2-Trichloroethane	1	ug/L
	Trichloroethene	1	ug/L
	Trichlorofluoromethane	1	ug/L
	Vinyl chloride	1	ug/L
Surrogates	1,4-Dichlorobutane		

GC Volatiles – Method 602
Reporting Limits (RL), Method Reference ^{1,2}

	Analyte	RL	Units
Water	Benzene	1	ug/L
	Chlorobenzene	1	ug/L
	1,2-Dichlorobenzene	1	ug/L
	1,3-Dichlorobenzene	1	ug/L
	1,4-Dichlorobenzene	1	ug/L
	Ethylbenzene	1	ug/L
	Toluene	1	ug/L
Surrogates	Trifluorotoluene		

**Total Petroleum Hydrocarbons, Diesel & Gasoline Range Organics
Reporting Limits (RL), Method Reference ^{1,2}**

	Analyte	RL	Units
Diesel Range Organics - Water	TPH (as Diesel)	100	ug/L
Surrogate	C9 (nonane)		
Diesel Range Organics - Solid	TPH (as Diesel)	10	mg/kg
Surrogate	C9 (nonane)		
Gasoline Range Organics - Water	Gasoline Range Organics	100	ug/L
	TPH (as Gasoline)	100	ug/L
	TPH (Purgeables)	100	ug/L
Gasoline Range Organics - Solid	Gasoline Range Organics	100	ug/kg
	TPH (as Gasoline)	100	ug/kg
	TPH (Purgeables)	100	ug/kg
Surrogate	TFT		
Gasoline Range Organics – Medium Level	TPH (as Gasoline) Solid	1000	ug/kg
	TPH (as Gasoline)	1000	ug/kg
	TPH (Purgeables)	1000	ug/kg
Surrogate	TFT		

Footnote:

¹ The latest MDLs, RLs, and Control Limits will be utilized at the time of sample analysis, and are available upon request.

² The compounds listed represent TAL/TCL compounds. Project specific or other regulatory lists may be available upon request.

Appendix 6. 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. (EPA-QAD)

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)

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 are 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 is 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)

Environmental Detection Limit (EDL):

The smallest level at which a radionuclide in an environmental medium can be unambiguously distinguished for a given confidence interval using a particular combination of sampling and measurement procedures, sample size, analytical detection limit, and processing procedure. The EDL shall be specified for the 0.95 or greater confidence interval. The EDL shall be established initially and verified annually for each test method and sample matrix. (NELAC Radioanalysis Subcommittee)

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 filling 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 shall 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 shall 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.

Manager (however named):

The individual designed as being responsible for the overall operation, all personnel, and the physical plant of the environmental laboratory. A supervisor may report to the manager. In some cases, the supervisor and the manager may be the same individual. (NELAC)

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 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 shall 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 shall 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) shall 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 shall 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 shall be analyzed at a minimum of 1 in 20 samples per matrix type per sample extraction or preparation method. The laboratory shall document their procedure to select the use of an appropriate type of duplicate. The selected sample(s) shall 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 shall 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)

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 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 shall 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 shall be kept on the measuring instrument's individual record.

Work Cell:

A well-defined group of analysts that together perform the method analysis. The members of the group and their specific functions within the work cell must be fully documented. (NELAC)

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 - Gas Chromatography/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

Appendix 7.

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	New Jersey	OH001
Connecticut	PH-0590	New York	10975
Florida	E87225	Pennsylvania	68-00340
Georgia	--	OVAP	CL0024
Illinois	200004	US ACE (Army)	--
Kansas	E-10336	USDA (Dept.of Agriculture)	S-62345
Kentucky Underground Storage Tank Program	0058	West Virginia	210
Minnesota	039-999-348	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.

Claims of Accreditation Status

TestAmerica North Canton 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 shall 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.

Should the company decide 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.

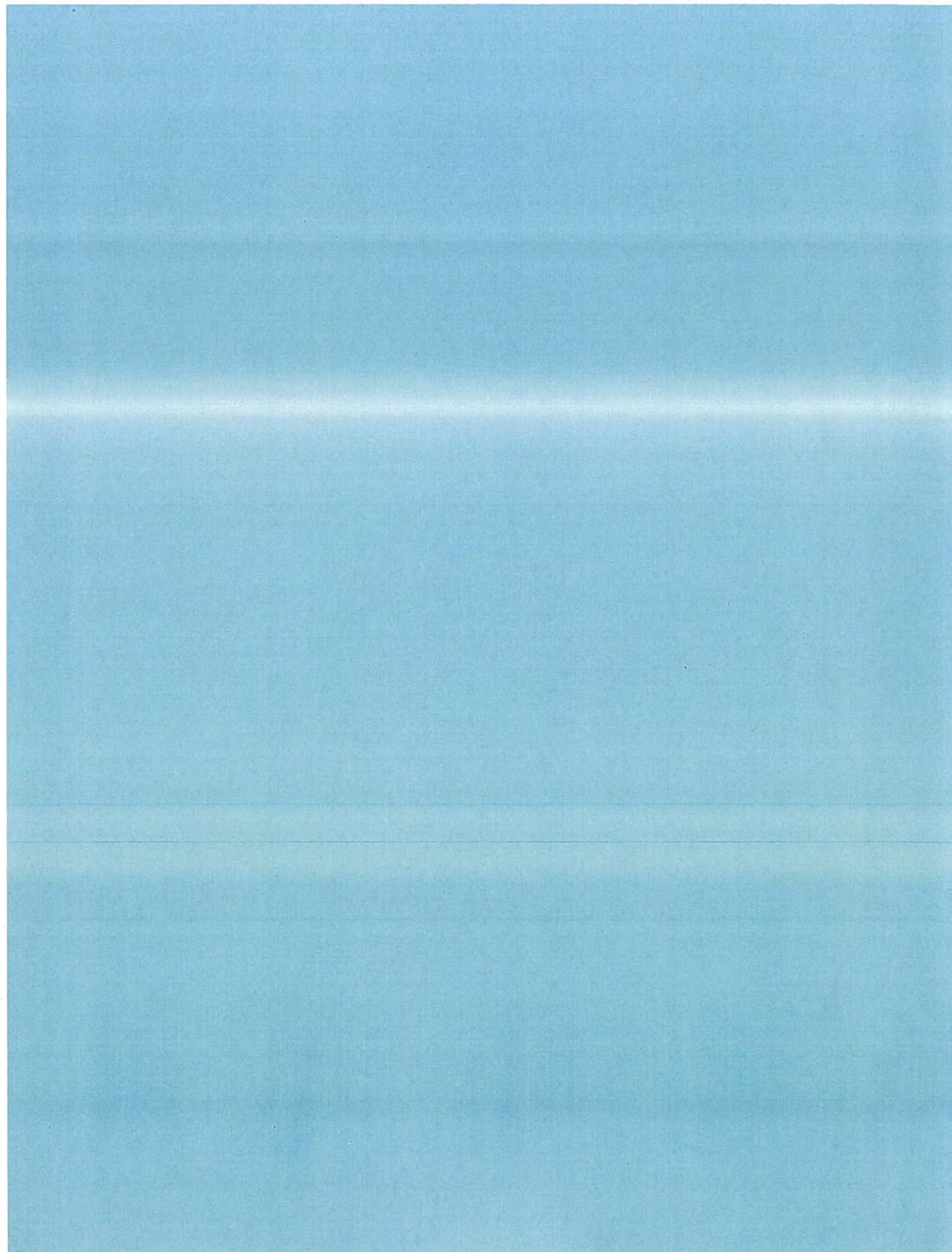
If accreditation is terminated or suspended, the laboratory will immediately cease to use the "Authority-accredited" wording, the logo, or the certificate number reference in any way and inform clients impacted by the change.

Appendix 8.

Data Qualifiers

Qualifier Organic	Qualifier Inorganic	Footnote
U	U	Analyte analyzed for but was not detected.
G	G	Elevated reporting limit. The reporting limit is elevated due to matrix interference.
J	B	Estimated result. Result is less than RL.
E	I	Estimated result. Result concentration exceeds the calibration range.
M	M	Matrix spike recovery outside of limits.
B	J	Method blank contamination. The associated method blank contains the target analyte at a reportable level.
P	*	Relative percent difference (RPD) is outside stated control limits.
a	N	Spiked analyte recovery is outside stated control limits.
*		Surrogate recovery is outside stated control limits.
PG		The percent difference between the original and confirmation analyses is greater than 40%.

This is not an exhaustive list of qualifiers. All qualifiers are defined on each data sheet. Client specific qualifiers may also be used, and would also be defined on the data sheet.



ATTACHMENT 3

LABORATORY REFERENCE DATA

ATTACHMENT 3

LABORATORY REFERENCE DATA

8260 water						LCS/D	LCS/D	LCS/D	MS/MSD	MS/MSD	MS/MSD
#	Compound RL	Units	MDL	Units	LCL	UCL	RPD	LCL	UCL	RPD	
11	Acetone	10 ug/L	1.1	ug/L	22	200	95	45	128	30	
196	Benzene	1 ug/L	0.13	ug/L	80	116	20	78	118	20	
323	Bromodich	1 ug/L	0.15	ug/L	87	130	30	80	146	30	
340	Bromoform	1 ug/L	0.64	ug/L	76	150	30	58	176	30	
343	Bromometl	1 ug/L	0.41	ug/L	64	129	30	55	145	30	
372	2-Butanon	10 ug/L	0.57	ug/L	28	237	65	71	123	30	
459	Carbon dis	1 ug/L	0.13	ug/L	73	139	30	69	138	41	
463	Carbon tet	1 ug/L	0.13	ug/L	75	149	30	63	176	30	
521	Chloroben	1 ug/L	0.15	ug/L	76	117	20	76	117	20	
535	Dibromoch	1 ug/L	0.18	ug/L	81	138	30	71	158	30	
550	Chloroetha	1 ug/L	0.29	ug/L	66	126	30	59	142	30	
569	Chloroform	1 ug/L	0.16	ug/L	84	128	30	83	141	30	
574	Chlorometl	1 ug/L	0.3	ug/L	48	123	30	40	137	39	
669	Cyclohexa	1 ug/L	0.12	ug/L	70	130	30	70	130	30	
539	1,2-Dibrom	2 ug/L	0.67	ug/L	70	130	30	70	130	30	
870	1,2-Dibrom	1 ug/L	0.24	ug/L	70	130	30	70	130	30	
904	1,2-Dichlor	1 ug/L	0.13	ug/L	70	130	30	70	130	30	
907	1,3-Dichlor	1 ug/L	0.14	ug/L	70	130	30	70	130	30	
910	1,4-Dichlor	1 ug/L	0.13	ug/L	70	130	30	70	130	30	
924	Dichlorodif	1 ug/L	0.31	ug/L	70	130	30	70	130	30	
933	1,1-Dichlor	1 ug/L	0.15	ug/L	86	123	30	88	127	30	
936	1,2-Dichlor	1 ug/L	0.22	ug/L	79	136	30	71	160	30	
948	cis-1,2-Dic	1 ug/L	0.17	ug/L	85	113	30	87	114	30	
950	trans-1,2-E	1 ug/L	0.19	ug/L	80	120	30	85	116	30	
943	1,1-Dichlor	1 ug/L	0.19	ug/L	63	130	20	62	130	20	
986	1,2-Dichlor	1 ug/L	0.18	ug/L	82	115	30	87	114	30	
998	cis-1,3-Dic	1 ug/L	0.14	ug/L	84	130	30	82	130	30	
1000	trans-1,3-E	1 ug/L	0.19	ug/L	84	130	30	73	147	30	
1332	Ethylbenze	1 ug/L	0.17	ug/L	86	116	30	86	132	30	
1515	2-Hexanon	10 ug/L	0.41	ug/L	35	200	52	81	128	30	
1578	Isopropylb	1 ug/L	0.13	ug/L	70	130	30	70	130	30	
1774	Methyl ace	10 ug/L	0.38	ug/L	70	130	30	70	130	30	
1799	Methylcycl	1 ug/L	0.13	ug/L	70	130	30	70	130	30	
1811	Methylene	1 ug/L	0.33	ug/L	78	118	30	82	115	30	
1845	4-Methyl-2	10 ug/L	0.32	ug/L	78	141	32	82	135	30	
2772	Methyl tert	5 ug/L	0.17	ug/L	70	130	30	70	130	30	
2355	Styrene	1 ug/L	0.11	ug/L	85	117	30	83	120	30	
2439	1,1,2,2-Tet	1 ug/L	0.18	ug/L	85	118	30	88	116	30	
2445	Tetrachlor	1 ug/L	0.29	ug/L	88	113	30	85	121	30	
2489	Toluene	1 ug/L	0.13	ug/L	74	119	20	70	119	20	
2515	1,2,4-Trich	1 ug/L	0.15	ug/L	70	130	30	70	130	30	
2518	1,1,1-Trich	1 ug/L	0.22	ug/L	78	140	30	71	162	30	
2522	1,1,2-Trich	1 ug/L	0.27	ug/L	83	122	30	86	129	30	
2525	Trichloroet	1 ug/L	0.17	ug/L	75	122	20	62	130	20	
1428	Trichlorofl	1 ug/L	0.21	ug/L	70	130	30	70	130	30	
2566	1,1,2-Trich	1 ug/L	0.28	ug/L	70	130	30	70	130	30	
2613	Vinyl chlori	1 ug/L	0.22	ug/L	61	120	30	88	126	30	
2627	Xylenes (tc	2 ug/L	0.28	ug/L	87	116	30	89	121	30	
337	4-Bromofluorobenzene				74	116	0	74	116	0	
2735	1,2-Dichloroethane-d4				61	128	0	61	128	0	
2740	Toluene-d8				76	110	0	76	110	0	
2863	Dibromofluoromethane				73	122	0	73	122	0	
8260 solid						LCS/D	LCS/D	LCS/D	MS/MSD	MS/MSD	MS/MSD
#	Compound RL	Units	MDL	Units	LCL	UCL	RPD	LCL	UCL	RPD	
11	Acetone	600 ug/kg	99	ug/kg	58	130	30	10	200	66	
196	Benzene	40 ug/kg	6.4	ug/kg	75	129	20	55	138	20	
323	Bromodich	80 ug/kg	12	ug/kg	72	125	30	47	131	51	
340	Bromoform	80 ug/kg	13	ug/kg	43	149	30	26	141	64	
343	Bromometl	200 ug/kg	25	ug/kg	24	152	30	15	152	72	
372	2-Butanon	600 ug/kg	49	ug/kg	27	200	46	21	195	60	
459	Carbon dis	200 ug/kg	20	ug/kg	50	137	30	27	149	73	
463	Carbon tet	40 ug/kg	12	ug/kg	57	137	30	32	143	68	

ATTACHMENT 3

LABORATORY REFERENCE DATA

521 Chloroben:	40 ug/kg	6.3 ug/kg	75	127	22	49	139	22
535 Dibromoch	40 ug/kg	6.9 ug/kg	49	135	30	44	135	61
550 Chloroetha	200 ug/kg	65 ug/kg	31	144	30	32	140	66
569 Chloroform	40 ug/kg	12 ug/kg	73	115	30	59	128	46
574 Chlorometl	200 ug/kg	5.2 ug/kg	15	136	30	28	130	81
669 Cyclohexai	960 ug/kg	7.7 ug/kg	50	150	20	50	150	20
539 1,2-Dibrom	200 ug/kg	60 ug/kg	50	150	30	50	150	20
870 1,2-Dibrom	200 ug/kg	9.9 ug/kg	50	150	30	50	150	20
904 1,2-Dichlor	80 ug/kg	17 ug/kg	50	150	30	50	150	20
907 1,3-Dichlor	80 ug/kg	7.2 ug/kg	50	150	20	50	150	20
910 1,4-Dichlor	80 ug/kg	7.9 ug/kg	50	150	20	50	150	20
924 Dichlorodif	80 ug/kg	5.4 ug/kg	50	150	20	50	150	20
933 1,1-Dichlor	40 ug/kg	7.3 ug/kg	77	119	30	56	130	54
936 1,2-Dichlor	40 ug/kg	9.3 ug/kg	78	121	30	56	126	38
948 cis-1,2-Dic	40 ug/kg	14 ug/kg	77	114	30	48	127	52
950 trans-1,2-C	40 ug/kg	11 ug/kg	68	117	30	47	127	58
943 1,1-Dichlor	40 ug/kg	8.3 ug/kg	55	142	27	43	147	27
986 1,2-Dichlor	40 ug/kg	7.4 ug/kg	78	116	30	54	125	43
998 cis-1,3-Dic	40 ug/kg	5.5 ug/kg	71	125	30	30	138	49
1000 trans-1,3-C	40 ug/kg	5.4 ug/kg	67	125	30	34	134	57
1332 Ethylbenze	40 ug/kg	6.8 ug/kg	79	114	30	36	133	72
1515 2-Hexanon	2000 ug/kg	24 ug/kg	29	200	41	20	190	70
1578 Isopropylb	200 ug/kg	5.1 ug/kg	50	150	20	50	150	20
1774 Methyl ace	960 ug/kg	52 ug/kg	50	150	20	50	150	20
1799 Methylcycl	960 ug/kg	5.7 ug/kg	50	150	20	50	150	20
1811 Methylene	200 ug/kg	99 ug/kg	58	130	30	45	129	49
1845 4-Methyl-2-	2000 ug/kg	11 ug/kg	68	142	60	42	143	60
2772 Methyl tert	200 ug/kg	10 ug/kg	70	130	30	70	130	30
2355 Styrene	40 ug/kg	28 ug/kg	80	114	30	23	136	65
2439 1,1,2,2-Tet	80 ug/kg	8.2 ug/kg	70	133	30	33	162	90
2445 Tetrachlor	40 ug/kg	9 ug/kg	72	120	30	31	137	81
2489 Toluene	80 ug/kg	9.1 ug/kg	71	130	24	46	147	24
2515 1,2,4-Trich	200 ug/kg	12 ug/kg	50	150	20	50	150	20
2518 1,1,1-Trich	40 ug/kg	9.5 ug/kg	67	123	30	48	132	57
2522 1,1,2-Trich	40 ug/kg	9.9 ug/kg	82	116	30	58	128	52
2525 Trichloroet	40 ug/kg	12 ug/kg	70	131	23	46	143	23
1428 Trichlorofl	80 ug/kg	8.3 ug/kg	50	150	20	50	150	20
2566 1,1,2-Trich	200 ug/kg	10 ug/kg	70	130	30	70	130	30
2613 Vinyl chlori	80 ug/kg	16 ug/kg	24	152	30	30	136	80
2627 Xylenes (tc	120 ug/kg	15 ug/kg	80	114	30	33	135	78
337 4-Bromofluorobenzene			47	158	0	47	158	0
2735 1,2-Dichloroethane-d4			61	130	0	61	130	0
2740 Toluene-d8			60	143	0	60	143	0
2863 Dibromofluoromethane			59	138	0	59	138	0

8270 solid

#	Compound RL	Units	MDL	Units	LCL	UCL	RPD	LCL	UCL	RPD
1	Acenaphth	330 ug/kg		1.3 ug/kg	46	110		30	10	200
5	Acenaphth	330 ug/kg		1.2 ug/kg	47	110		30	10	200
24	Acetophen	66.6 ug/kg		9 ug/kg	50	130		30	50	130
122	Anthracene	330 ug/kg		1.3 ug/kg	56	111		30	10	200
158	Atrazine	330 ug/kg		21 ug/kg	50	130		30	50	130
3398	Benzaldehy	330 ug/kg		21 ug/kg	10	130		30	10	130
202	Benzo(a)ai	330 ug/kg		0.95 ug/kg	58	111		30	10	200
205	Benzo(b)fl	330 ug/kg		1.2 ug/kg	43	124		30	10	200
208	Benzo(k)fl	330 ug/kg		1.7 ug/kg	38	122		30	10	200
210	Benzo(ghi)	330 ug/kg		1.3 ug/kg	44	120		30	10	200
211	Benzo(a)py	330 ug/kg		1.3 ug/kg	44	115		30	10	200
3474	1,1'-Biphen	330 ug/kg		23 ug/kg	50	130		30	50	130
289	bis(2-Chlor	330 ug/kg		22 ug/kg	42	110		30	36	110
293	bis(2-Chlor	330 ug/kg		2 ug/kg	41	110		30	32	118
302	bis(2-Ethyl	330 ug/kg		18 ug/kg	56	123		30	10	200
348	4-Bromoph	330 ug/kg		21 ug/kg	53	112		30	44	120
403	Butyl benz	330 ug/kg		19 ug/kg	57	121		30	43	138
5101	Caprolacta	330 ug/kg		37 ug/kg	50	130		30	50	130

ATTACHMENT 3

LABORATORY REFERENCE DATA

2751	Carbazole	330 ug/kg	19 ug/kg	56	115	30	10	162	30
518	4-Chloroar	330 ug/kg	17 ug/kg	25	110	30	11	110	30
578	4-Chloro-3	330 ug/kg	21 ug/kg	42	110	30	32	117	30
589	2-Chloron	330 ug/kg	22 ug/kg	46	110	30	40	110	30
600	2-Chlorop	330 ug/kg	26 ug/kg	39	110	30	32	110	30
602	4-Chlorop	330 ug/kg	24 ug/kg	53	110	30	47	116	30
633	Chrysene	330 ug/kg	0.9 ug/kg	56	111	30	10	200	30
860	Dibenz(a,h	330 ug/kg	1.3 ug/kg	45	122	30	10	200	30
863	Dibenzofur	330 ug/kg	20 ug/kg	50	110	30	10	200	30
891	Di-n-butyl	330 ug/kg	19 ug/kg	57	119	30	31	145	30
918	3,3'-Dichlor	1600 ug/kg	18 ug/kg	31	110	30	10	110	30
971	2,4-Dichlor	330 ug/kg	20 ug/kg	40	110	30	33	110	30
1082	Diethyl pht	330 ug/kg	19 ug/kg	55	114	30	48	118	30
1145	2,4-Dimeth	330 ug/kg	20 ug/kg	28	110	30	19	114	30
1149	Dimethyl pl	330 ug/kg	21 ug/kg	54	112	30	47	116	30
1167	4,6-Dinitro	1600 ug/kg	13 ug/kg	21	110	30	10	110	30
1187	2,4-Dinitro	1600 ug/kg	83 ug/kg	10	110	30	10	110	30
1191	2,4-Dinitro	330 ug/kg	18 ug/kg	55	116	30	42	118	30
1193	2,6-Dinitro	330 ug/kg	21 ug/kg	54	115	30	28	137	30
1162	Di-n-octyl	330 ug/kg	18 ug/kg	45	123	30	10	182	30
1414	Fluoranth	330 ug/kg	1.2 ug/kg	55	118	30	10	200	30
1417	Fluorene	330 ug/kg	1.2 ug/kg	51	110	30	10	187	30
1482	Hexachlor	330 ug/kg	2.1 ug/kg	51	110	30	37	122	30
1489	Hexachlor	330 ug/kg	26 ug/kg	39	110	30	30	110	30
1492	Hexachlor	1600 ug/kg	16 ug/kg	10	110	30	10	110	30
1497	Hexachlor	330 ug/kg	28 ug/kg	38	110	30	13	110	30
1535	Indeno(1,2	330 ug/kg	1.5 ug/kg	45	121	30	10	200	30
1566	Isophorone	330 ug/kg	21 ug/kg	46	117	30	32	129	30
1829	2-Methylna	330 ug/kg	1.5 ug/kg	46	110	30	10	200	30
1851	2-Methylph	330 ug/kg	28 ug/kg	36	110	30	19	124	30
1857	4-Methylph	330 ug/kg	22 ug/kg	40	110	30	27	116	30
1932	Naphthal	330 ug/kg	1.6 ug/kg	42	110	30	10	200	30
1960	2-Nitroanili	1600 ug/kg	22 ug/kg	47	124	30	31	141	30
1964	3-Nitroanili	1600 ug/kg	16 ug/kg	44	110	30	24	110	30
1968	4-Nitroanili	1600 ug/kg	26 ug/kg	50	110	30	23	124	30
1972	Nitrobenze	330 ug/kg	2.2 ug/kg	40	110	30	33	111	30
1998	2-Nitrophe	330 ug/kg	19 ug/kg	35	110	30	17	110	30
2001	4-Nitrophe	1600 ug/kg	110 ug/kg	24	117	30	10	125	30
2028	N-Nitrosod	330 ug/kg	21 ug/kg	54	112	30	10	169	30
2024	N-Nitrosod	330 ug/kg	23 ug/kg	40	114	30	30	121	30
3597	2,2'-oxybis	330 ug/kg	26 ug/kg	36	116	30	25	124	30
2118	Pentachlor	330 ug/kg	82 ug/kg	10	110	30	10	182	30
2154	Phenanthre	330 ug/kg	2 ug/kg	54	110	30	10	200	30
2155	Phenol	330 ug/kg	25 ug/kg	39	110	30	10	144	30
2252	Pyrene	330 ug/kg	1.1 ug/kg	58	113	30	10	200	30
2555	2,4,5-Trich	330 ug/kg	25 ug/kg	42	110	30	32	112	30
2559	2,4,6-Trich	330 ug/kg	21 ug/kg	37	110	30	22	110	30
1425	2-Fluorobiphenyl			34	110	0	34	110	0
1426	2-Fluorophenol			26	110	0	26	110	0
2512	2,4,6-Tribromophenol			10	118	0	10	118	0
2736	Nitrobenzene-d5			24	112	0	24	112	0
2737	Phenol-d5			28	110	0	28	110	0
2738	Terphenyl-d14			41	119	0	41	119	0

8270 water

#	Compound RL	Units	MDL	Units	LCL	UCL	RPD	LCL	UCL	RPD
1	Acenaphth	10 ug/L	0.054	ug/L	40	110	30	36	110	30
5	Acenaphth	10 ug/L	0.054	ug/L	43	110	30	39	110	30
24	Acetophen	10 ug/L	0.55	ug/L	50	130	30	50	130	30
122	Anthracene	10 ug/L	0.054	ug/L	54	114	30	46	110	30
158	Atrazine	10 ug/L	0.65	ug/L	50	130	30	50	130	30
3398	Benzaldeh	10 ug/L	0.75	ug/L	10	130	30	10	130	30
202	Benzo(a)ai	10 ug/L	0.052	ug/L	55	115	30	52	110	30
205	Benzo(b)flu	10 ug/L	0.049	ug/L	43	122	30	33	114	30
208	Benzo(k)flu	10 ug/L	0.049	ug/L	43	124	30	32	121	30

ATTACHMENT 3

LABORATORY REFERENCE DATA

210 Benzo(ghi)	10 ug/L	0.053 ug/L	45	120	30	34	116	30
211 Benzo(a)p	10 ug/L	0.048 ug/L	43	116	30	33	110	30
3474 1,1'-Biphe	10 ug/L	0.55 ug/L	50	130	30	50	130	30
289 bis(2-Chlor	10 ug/L	0.49 ug/L	39	110	30	35	110	30
293 bis(2-Chlor	10 ug/L	0.088 ug/L	34	113	30	27	110	30
302 bis(2-Ethyl	10 ug/L	0.88 ug/L	36	163	30	40	140	30
348 4-Bromoph	10 ug/L	0.52 ug/L	51	114	30	42	113	30
403 Butyl benz	10 ug/L	0.51 ug/L	53	126	30	51	121	30
5101 Caprolacta	10 ug/L	0.61 ug/L	50	130	30	50	130	30
2751 Carbazole	10 ug/L	0.54 ug/L	53	120	30	49	114	30
518 4-Chloroar	10 ug/L	0.56 ug/L	10	110	30	10	110	30
578 4-Chloro-3-	10 ug/L	0.41 ug/L	39	110	30	33	110	30
589 2-Chlorona	10 ug/L	0.62 ug/L	39	110	30	34	110	30
600 2-Chloroph	10 ug/L	0.039 ug/L	27	110	30	26	110	30
602 4-Chloroph	10 ug/L	0.55 ug/L	50	115	30	43	113	30
633 Chrysene	10 ug/L	0.048 ug/L	55	115	30	52	111	30
860 Dibenz(a,h	10 ug/L	0.039 ug/L	46	122	30	35	118	30
863 Dibenzofur	10 ug/L	0.54 ug/L	46	111	30	41	110	30
891 Di-n-butyl p	10 ug/L	0.61 ug/L	55	122	30	50	117	30
918 3,3'-Dichlor	50 ug/L	0.48 ug/L	19	110	30	10	110	30
971 2,4-Dichlor	10 ug/L	1.1 ug/L	33	110	30	30	110	30
1082 Diethyl pht	10 ug/L	0.63 ug/L	33	134	30	33	130	30
1145 2,4-Dimeth	10 ug/L	0.56 ug/L	12	110	30	11	110	30
1149 Dimethyl pi	10 ug/L	0.44 ug/L	15	143	30	36	124	30
1167 4,6-Dinitro	50 ug/L	0.27 ug/L	28	112	30	25	110	30
1187 2,4-Dinitro	50 ug/L	3.5 ug/L	17	112	30	11	119	30
1191 2,4-Dinitro	10 ug/L	0.4 ug/L	52	123	30	46	119	30
1193 2,6-Dinitro	10 ug/L	0.47 ug/L	52	119	30	48	115	30
1162 Di-n-octyl p	10 ug/L	0.39 ug/L	44	128	30	36	124	30
1414 Fluoranthene	10 ug/L	0.036 ug/L	54	122	30	53	111	30
1417 Fluorene	10 ug/L	0.043 ug/L	47	112	30	43	110	30
1482 Hexachloroc	10 ug/L	0.065 ug/L	51	112	30	40	113	30
1489 Hexachloroc	10 ug/L	0.51 ug/L	13	110	30	14	110	30
1492 Hexachloroc	10 ug/L	0.74 ug/L	10	110	30	10	110	30
1497 Hexachloroc	10 ug/L	0.58 ug/L	12	110	30	10	110	30
1535 Indeno(1,2	10 ug/L	0.065 ug/L	46	121	30	36	116	30
1566 Isophorone	10 ug/L	0.5 ug/L	44	128	30	34	125	30
1829 2-Methylna	10 ug/L	0.061 ug/L	35	110	30	35	110	30
1851 2-Methylph	10 ug/L	0.56 ug/L	30	110	30	26	110	30
1857 4-Methylph	10 ug/L	0.64 ug/L	32	110	30	25	110	30
1932 Naphthaler	10 ug/L	0.069 ug/L	31	110	30	32	110	30
1960 2-Nitroanili	50 ug/L	0.43 ug/L	43	130	30	31	129	30
1964 3-Nitroanili	50 ug/L	0.67 ug/L	45	116	30	23	112	30
1968 4-Nitroanili	50 ug/L	0.47 ug/L	45	120	30	26	115	30
1972 Nitrobenze	10 ug/L	0.053 ug/L	37	115	30	26	118	30
1998 2-Nitrophe	10 ug/L	1.3 ug/L	29	110	30	30	110	30
2001 4-Nitrophe	50 ug/L	0.63 ug/L	12	130	30	13	127	30
2028 N-Nitrosod	10 ug/L	0.46 ug/L	53	113	30	28	118	30
2024 N-Nitrosod	10 ug/L	0.53 ug/L	37	121	30	25	119	30
3597 2,2'-oxybis	10 ug/L	0.52 ug/L	25	128	30	13	124	30
2118 Pentachlor	10 ug/L	0.48 ug/L	26	110	30	23	110	30
2154 Phenanthre	10 ug/L	0.087 ug/L	52	114	30	47	110	30
2155 Phenol	10 ug/L	0.96 ug/L	14	112	30	16	110	30
2252 Pyrene	10 ug/L	0.048 ug/L	55	120	30	54	115	30
2555 2,4,5-Trich	10 ug/L	0.96 ug/L	39	110	30	36	110	30
2559 2,4,6-Trich	10 ug/L	1.4 ug/L	35	110	30	34	110	30
1425 2-Fluorobiphenyl			28	110	0	28	110	0
1426 2-Fluorophenol			10	110	0	10	110	0
2512 2,4,6-Tribromophenol			22	120	0	22	120	0
2736 Nitrobenzene-d5			27	111	0	27	111	0
2737 Phenol-d5			10	110	0	10	110	0
2738 Terphenyl-d14			37	119	0	37	119	0

8082 water

#	Compound RL	Units	MDL	Units	LCL	UCL	RPD	LCL	UCL	RPD

ATTACHMENT 3

LABORATORY REFERENCE DATA

2082 Aroclor 10'	0.2 ug/L	0.044 ug/L	44	119	30	10	166	30
2085 Aroclor 12'	0.2 ug/L	0.045 ug/L	0	0	0	0	0	0
2088 Aroclor 12'	0.2 ug/L	0.073 ug/L	0	0	0	0	0	0
2091 Aroclor 12'	0.2 ug/L	0.06 ug/L	0	0	0	0	0	0
2094 Aroclor 12'	0.2 ug/L	0.061 ug/L	0	0	0	0	0	0
2097 Aroclor 12'	0.2 ug/L	0.032 ug/L	0	0	0	0	0	0
2100 Aroclor 12'	0.2 ug/L	0.038 ug/L	41	118	30	21	140	30
2732 Decachlorobiphenyl			10	127	0	10	127	0
2739 Tetrachloro-m-xylene			27	130	0	27	130	0

8082 solid

#	Compound RL	Units	MDL	Units	LCL	UCL	RPD	LCL	UCL	RPD	
2082	Aroclor 10'	264 ug/kg		21 ug/kg	34	127		30	10	199	30
2085	Aroclor 12'	264 ug/kg		16 ug/kg	0	0		0	0	0	0
2088	Aroclor 12'	264 ug/kg		14 ug/kg	0	0		0	0	0	0
2091	Aroclor 12'	264 ug/kg		13 ug/kg	0	0		0	0	0	0
2094	Aroclor 12'	264 ug/kg		17 ug/kg	0	0		0	0	0	0
2097	Aroclor 12'	264 ug/kg		17 ug/kg	0	0		0	0	0	0
2100	Aroclor 12'	264 ug/kg		17 ug/kg	32	141		30	10	199	30
2732	Decachlorobiphenyl				10	199		0	10	199	0
2739	Tetrachloro-m-xylene				10	196		0	10	196	0

7471 solid

#	Compound RL	Units	MDL	Units	LCL	UCL	RPD	LCL	UCL	RPD
1701	Mercury	0.1 mg/kg		0.015 mg/kg	73	121	20	11	192	20

7470 water

#	Compound RL	Units	MDL	Units	LCL	UCL	RPD	LCL	UCL	RPD	
1701	Mercury	0.2 ug/L		0.12 ug/L		81	123	20	69	134	20

6010 water

#	Compound	RL	Units	MDL	Units	LCL	UCL	RPD	LCL	UCL	RPD	
88	Aluminum	200	ug/L		97 ug/L	80	120		20	75	125	20
128	Antimony	60	ug/L		1.8 ug/L	80	120		20	75	125	20
140	Arsenic	300	ug/L		3.2 ug/L	80	120		20	75	125	20
194	Barium	200	ug/L		0.67 ug/L	80	120		20	75	125	20
222	Beryllium	5	ug/L		0.46 ug/L	80	120		20	75	125	20
313	Boron	200	ug/L		34 ug/L	80	120		20	75	125	20
411	Cadmium	5	ug/L		0.66 ug/L	80	120		20	75	125	20
413	Calcium	5000	ug/L		130 ug/L	80	120		20	75	125	20
2952	Chromium	10	ug/L		2.2 ug/L	80	120		20	75	125	20
637	Cobalt	50	ug/L		1.7 ug/L	80	120		20	75	125	20
643	Copper	25	ug/L		4.5 ug/L	80	120		20	75	125	20
1539	Iron	100	ug/L		81 ug/L	77	122		20	75	125	20
1605	Lead	100	ug/L		1.9 ug/L	80	120		20	75	125	20
1618	Magnesium	5000	ug/L		34 ug/L	80	120		20	75	125	20
1659	Manganese	15	ug/L		0.41 ug/L	80	120		20	75	125	20
1906	Molybdenum	40	ug/L		1.3 ug/L	80	120		20	75	125	20
1956	Nickel	40	ug/L		3.2 ug/L	80	120		20	75	125	20
2214	Potassium	5000	ug/L		72 ug/L	80	120		20	75	125	20
2281	Selenium	250	ug/L		4.1 ug/L	80	120		20	75	125	20
2285	Silver	10	ug/L		2.2 ug/L	80	120		20	75	125	20
2315	Sodium	5000	ug/L		590 ug/L	80	120		20	75	125	20
2477	Thallium	200	ug/L		4.7 ug/L	80	120		20	75	125	20
2479	Tin	100	ug/L		3.3 ug/L	80	120		20	75	125	20
2482	Titanium	50	ug/L		5.3 ug/L	80	120		20	75	125	20
2607	Vanadium	50	ug/L		0.64 ug/L	80	120		20	75	125	20
2649	Zinc	20	ug/L		5 ug/L	80	120		20	75	125	20

6020 solid

#	Compound RL	Units	MDL	Units	LCL	UCL	RPD	LCL	UCL	RPD
88	Aluminum	5 mg/kg	1.3	mg/kg	80	120	20	70	130	20
128	Antimony	0.2 mg/kg	0.024	mg/kg	68	113	20	75	125	20
140	Arsenic	0.5 mg/kg	0.062	mg/kg	73	110	20	23	131	20
194	Barium	0.1 mg/kg	0.046	mg/kg	70	110	20	10	199	20
222	Beryllium	0.1 mg/kg	0.047	mg/kg	79	110	20	58	112	20
313	Boron	2 mg/kg	0.18	mg/kg	80	120	20	80	120	20

ATTACHMENT 3

LABORATORY REFERENCE DATA

411	Cadmium	0.1 mg/kg	0.0078 mg/kg	74	110	20	58	110	20
2952	Chromium	0.2 mg/kg	0.04 mg/kg	70	110	20	10	199	20
637	Cobalt	0.1 mg/kg	0.0038 mg/kg	74	110	20	55	110	20
643	Copper	0.2 mg/kg	0.043 mg/kg	73	110	20	10	199	20
1539	Iron	5 mg/kg	1 mg/kg	80	120	20	70	130	20
1605	Lead	0.1 mg/kg	0.013 mg/kg	75	110	20	10	199	20
1618	Magnesium	100 mg/kg	2.1 mg/kg	80	120	20	70	130	20
1659	Manganese	0.1 mg/kg	0.047 mg/kg	80	120	20	10	199	20
1906	Molybdenum	0.2 mg/kg	0.063 mg/kg	30	140	20	10	199	20
1956	Nickel	0.1 mg/kg	0.021 mg/kg	75	110	20	10	176	20
2214	Potassium	100 mg/kg	1.1 mg/kg	80	120	20	70	130	20
2281	Selenium	0.5 mg/kg	0.09 mg/kg	65	97	20	39	116	20
2285	Silver	0.1 mg/kg	0.016 mg/kg	60	114	20	75	125	20
2315	Sodium	100 mg/kg	2.4 mg/kg	80	120	20	70	130	20
2353	Strontium	1 mg/kg	0.063 mg/kg	80	120	20	52	110	20
2477	Thallium	0.1 mg/kg	0.013 mg/kg	71	110	20	62	110	20
2479	Tin	1 mg/kg	0.094 mg/kg	80	120	20	75	125	20
2602	Tungsten	1 mg/kg	0.041 mg/kg	75	125	20	80	120	20
2607	Vanadium	0.5 mg/kg	0.041 mg/kg	72	110	20	39	129	20
2649	Zinc	1 mg/kg	0.44 mg/kg	72	113	20	10	199	20
2651	Zirconium	2 mg/kg	0.2 mg/kg	80	120	20	80	120	20

6020 water

#	Compound RL	Units	MDL	Units	LCL	UCL	RPD	LCL	UCL	RPD
88	Aluminum	50 ug/L		19 ug/L	77	123	20	63	128	20
128	Antimony	2 ug/L		0.13 ug/L	57	110	20	44	153	20
140	Arsenic	5 ug/L		0.4 ug/L	86	118	20	82	123	20
194	Barium	1 ug/L		0.19 ug/L	83	110	20	45	144	20
222	Beryllium	1 ug/L		0.2 ug/L	84	120	20	77	124	20
313	Boron	20 ug/L		3.9 ug/L	80	120	20	80	120	20
411	Cadmium	1 ug/L		0.13 ug/L	89	114	20	78	117	20
413	Calcium	1000 ug/L		22 ug/L	80	120	20	70	130	20
2952	Chromium	2 ug/L		0.71 ug/L	81	110	20	72	110	20
637	Cobalt	1 ug/L		0.058 ug/L	82	113	20	67	114	20
643	Copper	2 ug/L		0.29 ug/L	82	113	20	60	123	20
1539	Iron	50 ug/L		26 ug/L	80	118	20	22	169	20
1605	Lead	1 ug/L		0.18 ug/L	84	113	20	73	115	20
1618	Magnesium	1000 ug/L		17 ug/L	80	120	20	70	130	20
1659	Manganese	1 ug/L		0.83 ug/L	78	111	20	10	172	20
1906	Molybdenum	2 ug/L		0.093 ug/L	62	111	20	80	110	20
1956	Nickel	2 ug/L		0.2 ug/L	80	111	20	72	111	20
2214	Potassium	1000 ug/L		8.3 ug/L	80	120	20	70	130	20
2281	Selenium	5 ug/L		1.2 ug/L	90	128	20	72	148	20
2285	Silver	1 ug/L		0.08 ug/L	83	111	20	10	139	20
2315	Sodium	1000 ug/L		6.9 ug/L	80	120	20	80	120	20
2353	Strontium	10 ug/L		0.33 ug/L	82	110	20	20	192	20
2477	Thallium	1 ug/L		0.14 ug/L	82	113	20	69	117	20
2479	Tin	10 ug/L		0.31 ug/L	77	110	20	50	117	20
2482	Titanium	10 ug/L		0.64 ug/L	80	120	20	75	125	20
2602	Tungsten	10 ug/L		0.37 ug/L	78	110	20	43	133	20
2607	Vanadium	20 ug/L		0.44 ug/L	82	110	20	70	112	20
2649	Zinc	20 ug/L		2.3 ug/L	90	129	20	49	156	20
2651	Zirconium	20 ug/L		2.5 ug/L	80	120	20	80	120	20

8260 waste

#	Compound	RL	Units	MDL	Units	LCL	UCL	RPD	LCL	UCL	RPD	
11	Acetone	2500	ug/kg		99 ug/kg	58	130	130	30	10	200	66
196	Benzene	620	ug/kg		6.4 ug/kg	75	129	129	20	55	138	20
323	Bromodich	620	ug/kg		12 ug/kg	72	125	125	30	47	131	51
340	Bromoform	620	ug/kg		13 ug/kg	43	149	149	30	26	141	64
343	Bromomet	1200	ug/kg		25 ug/kg	24	152	152	30	15	152	72
372	2-Butanone	2500	ug/kg		49 ug/kg	27	200	200	46	21	195	60
459	Carbon dis	620	ug/kg		20 ug/kg	50	137	137	30	27	149	73
463	Carbon tet	620	ug/kg		12 ug/kg	57	137	137	30	32	143	68
521	Chloroben	620	ug/kg		6.3 ug/kg	75	127	127	22	49	139	22
535	Dibromoch	620	ug/kg		6.9 ug/kg	49	135	135	30	44	135	61

ATTACHMENT 3

LABORATORY REFERENCE DATA

550	Chloroetha	1200 ug/kg	65 ug/kg	31	144	30	32	140	66
569	Chloroform	620 ug/kg	12 ug/kg	73	115	30	59	128	46
574	Chloromett	1200 ug/kg	5.2 ug/kg	15	136	30	28	130	81
669	Cyclohexa	2500 ug/kg	7.7 ug/kg	50	150	20	50	150	20
539	1,2-Dibrom	1200 ug/kg	60 ug/kg	50	150	30	50	150	20
870	1,2-Dibrom	620 ug/kg	9.9 ug/kg	50	150	30	50	150	20
904	1,2-Dichlor	1200 ug/kg	17 ug/kg	50	150	30	50	150	20
907	1,3-Dichlor	1200 ug/kg	7.2 ug/kg	50	150	20	50	150	20
910	1,4-Dichlor	1200 ug/kg	7.9 ug/kg	50	150	20	50	150	20
924	Dichlorodif	1200 ug/kg	5.4 ug/kg	50	150	20	50	150	20
933	1,1-Dichlor	620 ug/kg	7.3 ug/kg	77	119	30	56	130	54
936	1,2-Dichlor	620 ug/kg	9.3 ug/kg	78	121	30	56	126	38
948	cis-1,2-Dic	310 ug/kg	14 ug/kg	77	114	30	48	127	52
950	trans-1,2-E	310 ug/kg	11 ug/kg	68	117	30	47	127	58
943	1,1-Dichlor	620 ug/kg	8.3 ug/kg	55	142	27	43	147	27
986	1,2-Dichlor	620 ug/kg	7.4 ug/kg	78	116	30	54	125	43
998	cis-1,3-Dic	620 ug/kg	5.5 ug/kg	71	125	30	30	138	49
1000	trans-1,3-E	620 ug/kg	5.4 ug/kg	67	125	30	34	134	57
1332	Ethylbenze	620 ug/kg	6.8 ug/kg	79	114	30	36	133	72
1515	2-Hexanon	2500 ug/kg	24 ug/kg	29	200	41	20	190	70
1578	Isopropylb	1200 ug/kg	5.1 ug/kg	50	150	20	50	150	20
1774	Methyl ace	1200 ug/kg	52 ug/kg	50	150	20	50	150	20
1799	Methylcycl	620 ug/kg	8.92 ug/kg	50	150	20	50	150	20
1811	Methylene	620 ug/kg	99 ug/kg	58	130	30	45	129	49
1845	4-Methyl-2	2500 ug/kg	11 ug/kg	68	142	30	42	143	60
2772	Methyl tert	2500 ug/kg	10 ug/kg	70	130	30	70	130	30
2355	Styrene	620 ug/kg	28 ug/kg	80	114	30	23	136	65
2439	1,1,2,2-Tet	620 ug/kg	8.2 ug/kg	70	133	30	33	162	90
2445	Tetrachlor	620 ug/kg	9 ug/kg	72	120	30	31	137	81
2489	Toluene	620 ug/kg	9.1 ug/kg	71	130	24	46	147	24
2515	1,2,4-Trich	1200 ug/kg	12 ug/kg	50	150	20	50	150	20
2518	1,1,1-Trich	620 ug/kg	9.5 ug/kg	67	123	30	48	132	57
2522	1,1,2-Trich	620 ug/kg	9.9 ug/kg	82	116	30	58	128	52
2525	Trichloroet	620 ug/kg	12 ug/kg	70	131	23	46	143	23
1428	Trichlorofl	1200 ug/kg	8.3 ug/kg	50	150	20	50	150	20
2566	1,1,2-Trich	2500 ug/kg	10 ug/kg	70	130	30	70	130	30
2613	Vinyl chlori	1200 ug/kg	16 ug/kg	24	152	30	30	136	80
2627	Xylenes (tc	620 ug/kg	15 ug/kg	80	114	30	33	135	78
337	4-Bromofluorobenzene			47	158	0	47	158	0
2735	1,2-Dichloroethane-d4			61	130	0	61	130	0
2740	Toluene-d8			60	143	0	60	143	0
2863	Dibromofluoromethane			59	138	0	59	138	0

8270 waste

#	Compound RL	Units	MDL	Units	LCL	UCL	RPD	LCL	UCL	RPD
1	Acenaphth	20000 ug/kg	55.2	ug/kg	52	110	30	10	200	30
5	Acenaphth	20000 ug/kg	84	ug/kg	56	110	30	10	200	30
24	Acetophen	20000 ug/kg	330	ug/kg	0	0	0	0	0	0
122	Anthracene	20000 ug/kg	138	ug/kg	59	110	30	10	200	30
158	Atrazine	20000 ug/kg	660	ug/kg	0	0	0	0	0	0
3398	Benzaldehy	20000 ug/kg	414	ug/kg	10	130	30	10	130	30
202	Benzo(a)ai	20000 ug/kg	84	ug/kg	54	110	30	10	200	30
205	Benzo(b)fl	20000 ug/kg	138	ug/kg	50	110	30	10	200	30
208	Benzo(k)fl	20000 ug/kg	138	ug/kg	46	110	30	10	200	30
210	Benzo(ghi)	20000 ug/kg	108	ug/kg	49	110	30	10	200	30
211	Benzo(a)p	20000 ug/kg	138	ug/kg	45	110	30	10	200	30
3474	1,1'-Biphe	20000 ug/kg	432	ug/kg	0	0	0	0	0	0
289	bis(2-Chlor	20000 ug/kg	960	ug/kg	54	110	30	36	110	30
293	bis(2-Chlor	20000 ug/kg	246	ug/kg	41	124	30	32	118	30
302	bis(2-Ethyl	20000 ug/kg	960	ug/kg	52	113	30	10	200	30
348	4-Bromoph	20000 ug/kg	282	ug/kg	59	110	30	44	120	30
403	Butyl benz	20000 ug/kg	222	ug/kg	58	110	30	43	138	30
5101	Caprolacta	20000 ug/kg	468	ug/kg	0	0	0	0	0	0
2751	Carbazole	20000 ug/kg	558	ug/kg	59	110	30	10	162	30
518	4-Chloroar	20000 ug/kg	312	ug/kg	24	110	30	11	110	30

ATTACHMENT 3

LABORATORY REFERENCE DATA

578	4-Chloro-3	20000 ug/kg	3060 ug/kg	51	110	30	32	117	30
589	2-Chlorone	20000 ug/kg	378 ug/kg	56	110	30	40	110	30
600	2-Chloroph	20000 ug/kg	216 ug/kg	51	110	30	32	110	30
602	4-Chloroph	20000 ug/kg	216 ug/kg	59	110	30	47	116	30
633	Chrysene	20000 ug/kg	59.4 ug/kg	54	110	30	10	200	30
860	Dibenz(a,h	20000 ug/kg	90 ug/kg	47	110	30	10	200	30
863	Dibenzofur	20000 ug/kg	49.8 ug/kg	57	110	30	10	200	30
891	Di-n-butyl p	20000 ug/kg	300 ug/kg	60	110	30	31	145	30
918	3,3'-Dichlor	96000 ug/kg	294 ug/kg	36	110	30	10	110	30
971	2,4-Dichlor	20000 ug/kg	318 ug/kg	52	110	30	33	110	30
1082	Diethyl pht	20000 ug/kg	372 ug/kg	57	110	30	48	118	30
1145	2,4-Dimeth	20000 ug/kg	408 ug/kg	44	110	30	19	114	30
1149	Dimethyl pl	20000 ug/kg	384 ug/kg	59	110	30	47	116	30
1167	4,6-Dinitro-	96000 ug/kg	2820 ug/kg	35	110	30	10	110	30
1187	2,4-Dinitro	96000 ug/kg	2460 ug/kg	10	148	30	10	110	30
1191	2,4-Dinitro	20000 ug/kg	342 ug/kg	61	110	30	42	118	30
1193	2,6-Dinitro	20000 ug/kg	348 ug/kg	60	110	30	28	137	30
1162	Di-n-octyl p	20000 ug/kg	660 ug/kg	55	110	30	10	182	30
1414	Fluoranthene	20000 ug/kg	55.8 ug/kg	58	110	30	10	200	30
1417	Fluorene	20000 ug/kg	78 ug/kg	54	110	30	10	187	30
1482	Hexachloro	20000 ug/kg	84 ug/kg	58	110	30	37	122	30
1489	Hexachloro	20000 ug/kg	150 ug/kg	59	110	30	30	110	30
1492	Hexachloro	96000 ug/kg	174 ug/kg	31	110	30	10	110	30
1497	Hexachloro	20000 ug/kg	294 ug/kg	60	110	30	13	110	30
1535	Indeno(1,2	20000 ug/kg	120 ug/kg	44	110	30	10	200	30
1566	Isophorone	20000 ug/kg	210 ug/kg	52	128	30	32	129	30
1829	2-Methylna	20000 ug/kg	59.4 ug/kg	61	110	30	10	200	30
1851	2-Methylph	20000 ug/kg	402 ug/kg	50	110	30	19	124	30
1857	4-Methylph	20000 ug/kg	348 ug/kg	53	110	30	27	116	30
1932	Naphthaler	20000 ug/kg	53.4 ug/kg	55	110	30	10	200	30
1960	2-Nitroanili	96000 ug/kg	306 ug/kg	55	110	30	31	141	30
1964	3-Nitroanili	96000 ug/kg	192 ug/kg	47	110	30	27	110	30
1968	4-Nitroanili	96000 ug/kg	216 ug/kg	53	110	30	23	124	30
1972	Nitrobenze	20000 ug/kg	384 ug/kg	50	115	30	33	111	30
1998	2-Nitrophe	20000 ug/kg	204 ug/kg	48	110	30	17	110	30
2001	4-Nitrophe	96000 ug/kg	4860 ug/kg	43	110	30	10	125	30
2028	N-Nitrosod	20000 ug/kg	246 ug/kg	59	110	30	10	169	30
2024	N-Nitrosod	20000 ug/kg	456 ug/kg	42	131	30	30	121	30
3597	2,2'-oxybis	20000 ug/kg	312 ug/kg	39	128	30	25	124	30
2118	Pentachlor	20000 ug/kg	2700 ug/kg	42	110	30	10	182	30
2154	Phenanthro	20000 ug/kg	66 ug/kg	58	110	30	10	200	30
2155	Phenol	20000 ug/kg	342 ug/kg	47	110	30	10	144	30
2252	Pyrene	20000 ug/kg	60 ug/kg	56	110	30	10	200	30
2555	2,4,5-Trich	20000 ug/kg	294 ug/kg	49	110	30	32	112	30
2559	2,4,6-Trich	20000 ug/kg	408 ug/kg	51	110	30	22	110	30
1425	2-Fluorobiphenyl			44	110	0	44	110	0
1426	2-Fluorophenol			49	110	0	49	110	0
2512	2,4,6-Tribromophenol			16	124	0	16	124	0
2736	Nitrobenzene-d5			53	110	0	53	110	0
2737	Phenol-d5			47	110	0	47	110	0
2738	Terphenyl-d14			36	128	0	36	128	0

8082 waste

#	Compound RL	Units	MDL	Units	LCL	UCL	RPD	LCL	UCL	RPD
2082	Aroclor 10	1000 ug/kg		190 ug/kg	34	127	30	10	199	30
2085	Aroclor 12	1000 ug/kg		220 ug/kg	0	0	0	0	0	0
2088	Aroclor 12	1000 ug/kg		170 ug/kg	0	0	0	0	0	0
2091	Aroclor 12	1000 ug/kg		290 ug/kg	0	0	0	0	0	0
2094	Aroclor 12	1000 ug/kg		200 ug/kg	0	0	0	0	0	0
2097	Aroclor 12	1000 ug/kg		120 ug/kg	0	0	0	0	0	0
2100	Aroclor 12	1000 ug/kg		130 ug/kg	32	141	30	10	199	30
2732	Decachlorobiphenyl				10	199	0	10	199	0
2739	Tetrachloro-m-xylene				10	196	0	10	196	0

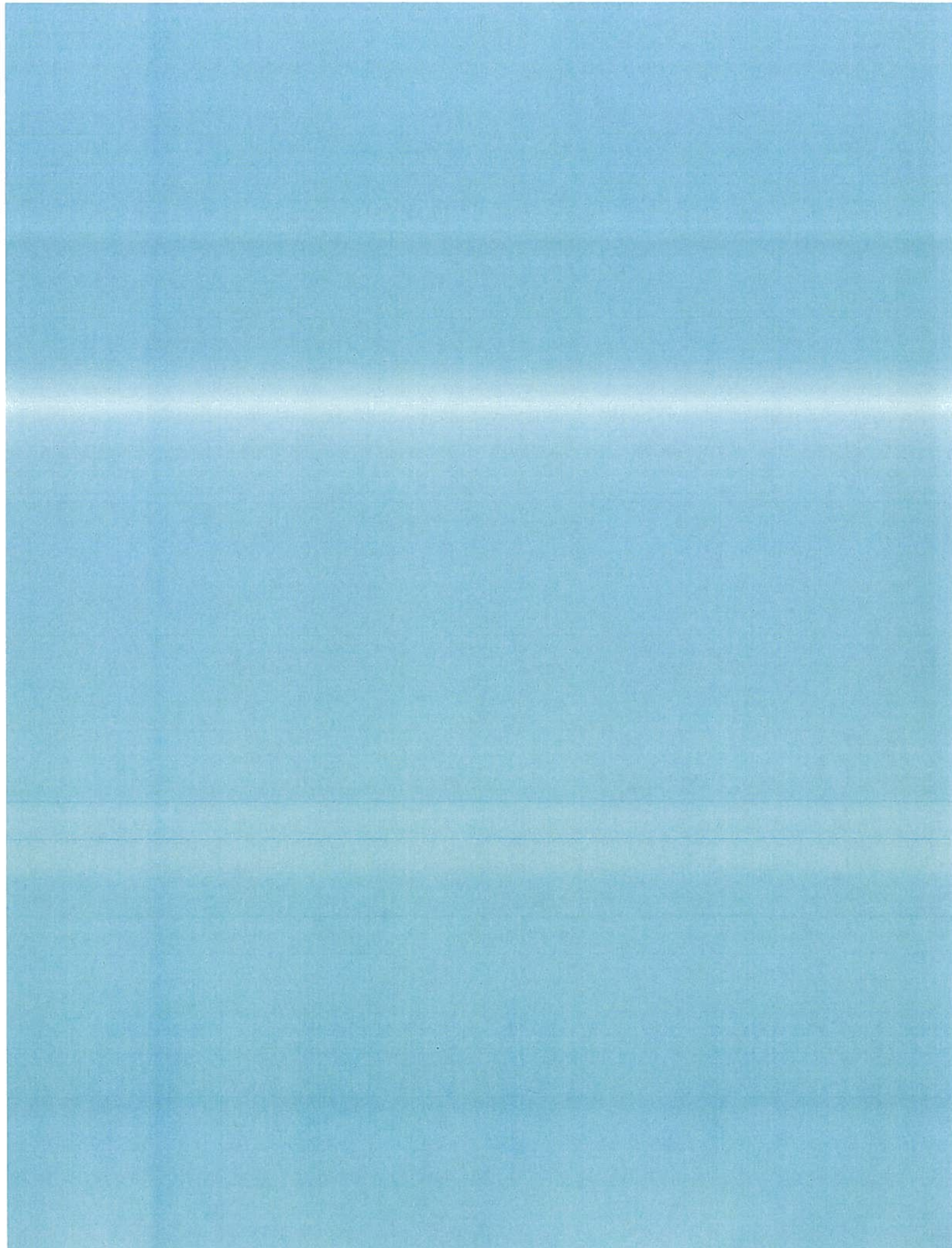
6010 waste

#	Compound RL	Units	MDL	Units	LCL	UCL	RPD	LCL	UCL	RPD

ATTACHMENT 3

LABORATORY REFERENCE DATA

88 Aluminum	20 mg/kg	9.6 mg/kg	80	120	20	75	125	20			
128 Antimony	6 mg/kg	0.39 mg/kg	80	120	20	75	125	20			
140 Arsenic	30 mg/kg	0.3 mg/kg	80	120	20	75	125	20			
194 Barium	20 mg/kg	0.071 mg/kg	80	120	20	75	125	20			
222 Beryllium	0.5 mg/kg	0.043 mg/kg	80	120	20	75	125	20			
313 Boron	20 mg/kg	3.4 mg/kg	80	120	20	75	125	20			
411 Cadmium	0.5 mg/kg	0.036 mg/kg	80	120	20	75	125	20			
413 Calcium	500 mg/kg	16 mg/kg	80	120	20	75	125	20			
2952 Chromium	1 mg/kg	0.2 mg/kg	80	120	20	75	125	20			
637 Cobalt	5 mg/kg	0.16 mg/kg	80	120	20	75	125	20			
643 Copper	2.5 mg/kg	0.74 mg/kg	80	120	20	75	125	20			
1539 Iron	10 mg/kg	4.9 mg/kg	73	137	20	75	125	20			
1605 Lead	10 mg/kg	0.19 mg/kg	80	120	20	75	125	20			
1618 Magnesium	500 mg/kg	5.1 mg/kg	80	120	20	75	125	20			
1659 Manganese	1.5 mg/kg	0.074 mg/kg	80	120	20	75	125	20			
1906 Molybdenum	4 mg/kg	0.27 mg/kg	80	120	20	75	125	20			
1956 Nickel	4 mg/kg	0.27 mg/kg	80	120	20	75	125	20			
2200 Phosphorus	30 mg/kg		0	0	0	0	0	0			
2214 Potassium	500 mg/kg	6.2 mg/kg	80	120	20	75	125	20			
2281 Selenium	25 mg/kg	0.45 mg/kg	80	120	20	75	125	20			
2285 Silver	1 mg/kg	0.1 mg/kg	80	120	20	75	125	20			
2315 Sodium	500 mg/kg	66 mg/kg	80	120	20	75	125	20			
2477 Thallium	200 mg/kg	0.55 mg/kg	80	120	20	75	125	20			
2479 Tin	10 mg/kg	0.5 mg/kg	80	120	20	75	125	20			
2482 Titanium	5 mg/kg	0.57 mg/kg	80	120	20	75	125	20			
2607 Vanadium	5 mg/kg	0.12 mg/kg	80	120	20	75	125	20			
2649 Zinc	2 mg/kg	1 mg/kg	80	120	20	75	125	20			
7471 waste											
#	Compound RL	Units	MDL	Units	LCL	UCL	RPD	LCL	UCL	RPD	
	1701 Mercury	0.1 mg/kg		0.015 mg/kg		81	116	20	30	134	20
9012 waste											
#	Compound RL	Units	MDL	Units	LCL	UCL	RPD	LCL	UCL	RPD	
	667 Total Cyan	0.5 mg/kg		0.1 mg/kg		65	124	20	50	134	20
DRO Waste											
#	Compound RL	Units	MDL	Units	LCL	UCL	RPD	LCL	UCL	RPD	
	5281 TPH C10-C	1000 mg/kg		2.8 mg/kg		0	0	0	0	0	0
	3052 TPH (as Di	1000 mg/kg		2.8 mg/kg		47	138	30	10	199	30
	2976 C9 (nonane)					10	110	0	10	110	0



ATTACHMENT 4

FIELD AUDIT CHECKLIST

ATTACHMENT 4

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.

ATTACHMENT 4

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?

ATTACHMENT 4

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.

ATTACHMENT 4

FIELD AUDIT CHECKLIST

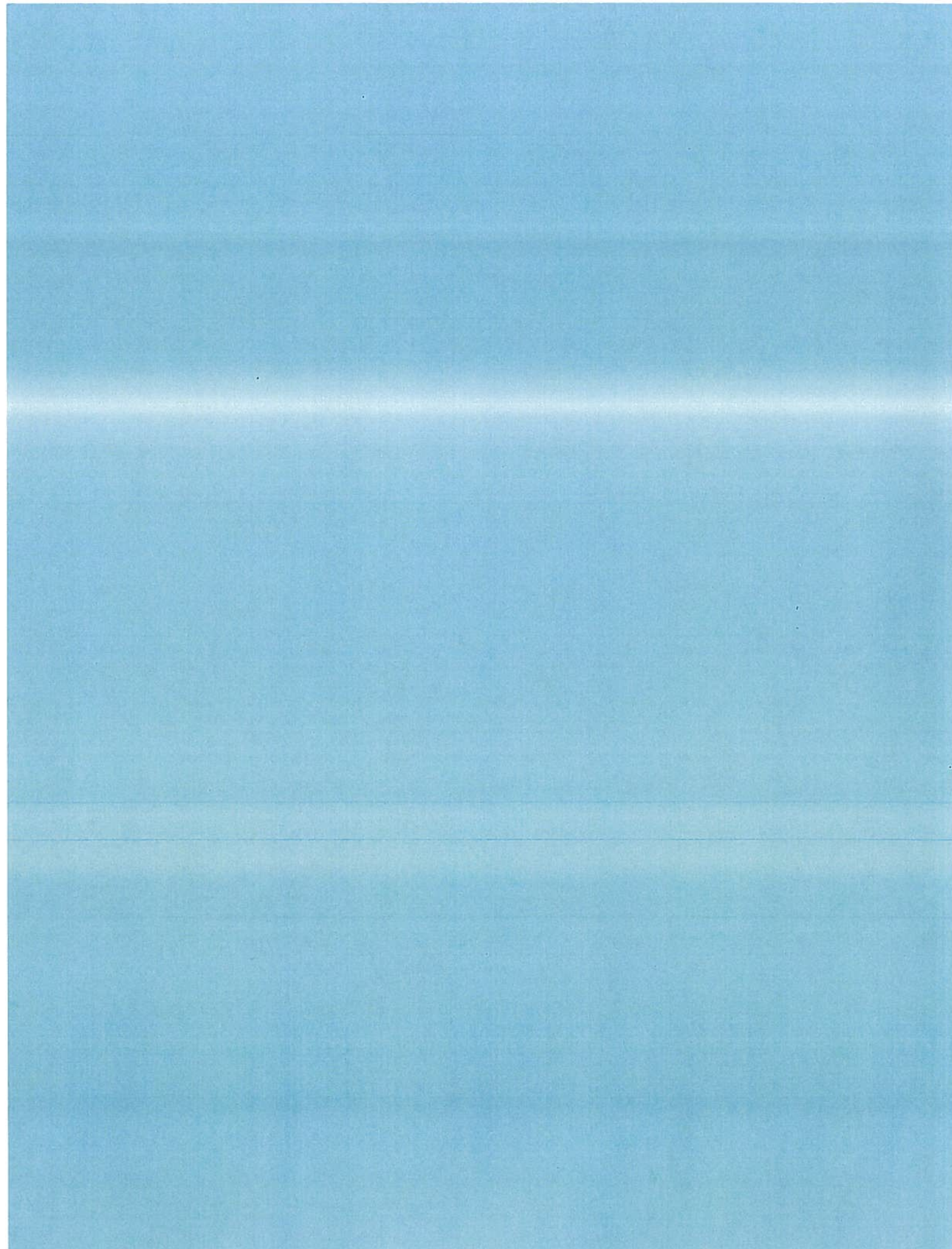
Debriefing with Project Manager

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.



ATTACHMENT 5

LABORATORY AUDIT CHECKLIST

ATTACHMENT 5

LABORATORY AUDIT CHECKLIST

SIGNATURE OF AUDITOR _____ DATE OF AUDIT _____

PROJECT MANAGER _____ PROJECT NO. _____

PROJECT LOCATION _____

TYPE OF INVESTIGATION _____
(authority, agency)

The enclosed laboratory audit checklists are organized into the categories by laboratory functional area. The attached audit checklists have been numbered according to the following outline:

- 1.0 Sample Management
- 2.0 Organic Sample Preparation
- 3.0 Gas Chromatography
- 4.0 Mass Spectrometry
- 5.0 Metals Preparation
- 6.0 Metals Analysis
- 7.0 Wet Chemistry
- 8.0 Report Generation
- 9.0 Computer Systems

Special Checklists

- Form A Laboratory Temperature Monitoring Checklist
- Form B Laboratory Notebook Checklist
- Form C Laboratory Balance Checklist
- Form D Reagent and Analytical Standards Checklist
- Form E Laboratory Safety Checklist

ATTACHMENT 5

LABORATORY AUDIT CHECKLIST

1.0 Sample Management

Shipment of Sample Bottles and Coolers

Check off which of the following items are utilized in this area and complete the associated forms.

☐	☐	Refrigerators, freezers, ovens,	Complete Form A
☐	☐	Logbooks	Complete Form B
☐	☐	Laboratory Balances	Complete Form C
☐	☐	Reagents and Standards	Complete Form D
☐	☐	Safety Equipment	Complete Form E

Y	N	Item	Comments
☐	☐	Is there a SOP describing preparation and shipment of sample coolers?	
☐	☐	Are sample bottles precleaned and is cleaning documented?	
☐	☐	Are cooler shipment records maintained?	
☐	☐	Are coolers and bottles sent out under chain-of-custody?	
☐	☐	Are preservatives provided with the cooler?	
☐	☐	Are sample collectors supplied with preservatives and sampling instructions for their use?	
☐	☐	Is there a SOP describing the amount and concentration of preservation agents added to bottles?	
☐	☐	Are chain of custody forms provided along with instructions for proper completion?	
☐	☐	Are trip blanks included with the cooler?	
☐	☐	If so, is the purity of the trip blank water checked before shipment and can it be documented?	
☐	☐	Does the laboratory only utilize new sample containers?	
☐	☐		
☐	☐		

ATTACHMENT 5

LABORATORY AUDIT CHECKLIST

1.0 Sample Management (continued)

Receipt and Storage of Samples

Check off which of the following items are utilized in this area and complete the associated forms.		
☐	Refrigerators, freezers, ovens,	Complete Form A
☐	Logbooks	Complete Form B
☐	Laboratory Balances	Complete Form C
☐	Reagents and Standards	Complete Form D
☐	Safety Equipment	Complete Form E

Y	N	Item	Comment
		Do the laboratory personnel indicate a familiarity with the requirements set forth in the laboratory QA program?	
		Is there a written SOP readily available to the login personnel?	
		Is the SOP followed as written?	
		If discrepancies are noted between COC records and the samples, are they documented and appropriate personnel notified?	
		Is the pH checked on all appropriate incoming samples and documented?	
		Is the pH check done in such a manner that avoids sample contamination?	
		Is HCl preservation utilized for volatile sample preservation? If not, is a 7-day hold time being applied?	
		Are incoming volatile vials checked for the presence of air bubbles?	
		If air bubbles are detected, is the client notified?	
		If there a mechanism to receive samples after normal work hours and weekends?	
		Is the filing system for original custody forms and paperwork traceable?	
		Is a fume hood available for opening of hazardous samples?	
		Are eyewashes and/or showers available in the immediate area?	
		Is there a central log-in notebook and is it document controlled?	
		Are unique laboratory sample identification numbers assigned and properly recorded on sample container labels and sample log-in	

ATTACHMENT 5

LABORATORY AUDIT CHECKLIST

		documentation?	

1.0 Sample Management (continued)

Y	N	Item	Comment
		Is there a SOP describing how the lab handles samples with short holding times?	
		Are samples checked for proper preservation upon receipt? (Required by NJ)	
		If samples are not properly preserved, is there a SOP describing how it is handled?	
		Are files maintained for log-in documentation?	
		Are all samples and extracts stored in a secure manner? (Locked refrigerators and freezers)	
		Are all samples and extracts stored in separate storage units?	
		Is there limited access to sample and extract storage areas?	
		Is there an internal custody procedure for samples and extracts and is it documented in a SOP? If so, where are records filed?	
		Is there a procedure for documenting where samples and extracts are stored?	
		Are volatile samples stored separately from other samples or extracts?	
		Are volatile water samples stored separately from soil volatile samples?	
		Are refrigerator holding blanks utilized to monitor potential sample contamination in each volatile sample storage unit?	
		Are standards or reagents stored with samples or extracts?	
		Are samples maintained in a cooled condition until holding time expires?	
		Are archived samples stored in a secure manner?	
		Is there a policy describing how long samples are held after a report is issued?	
		Are samples being held after report issuance maintained in a cooled condition?	
		Is there a SOP describing the archival and disposal of samples and sample extracts?	

ATTACHMENT 5

LABORATORY AUDIT CHECKLIST

1.0 Sample Management (continued)

Y	N	Item	Comment
		Does the disposal SOP describe how the lab disposes of samples of unknown content?	
		Is there a record of when every sample has been discarded?	
		Do supervisory personnel review the paperwork in the dept. and is this review documented?	

ATTACHMENT 5

LABORATORY AUDIT CHECKLIST

2.0 Organic Sample Preparation

Check off which of the following items are utilized in this area and complete the associated forms.

☐	Refrigerators, freezers, ovens,	Complete Form A
☐	Logbooks	Complete Form B
☐	Laboratory Balances	Complete Form C
☐	Reagents and Standards	Complete Form D
☐	Safety Equipment	Complete Form E

Y	N	Item	Comment
		Are SOPs available detailing the procedures used in this area?	Obtain a listing of all procedures performed in the area. Compare the list with available SOPs and estimate a percentage complete below. Percent Compliance _____

Are the following QC samples processed with each analytical batch?

☐	☐	Method Blank	
☐	☐	QC Check Sample	
☐	☐	Matrix Spike	
☐	☐	Matrix Spike Duplicate	
☐	☐	Are the solvent storage cabinets properly vented as appropriate to avoid possible laboratory contamination?	
☐	☐	Are gas lines checked for leaks each time a tank is changed?	
☐	☐	If the lab utilizes desiccators, do they appear to be properly maintained?	
☐	☐	Is there an SOP for glassware washing and is it posted in the washing area?	
☐	☐	Are operating manuals available to the analysts for all major equipment?	
☐	☐	Is there a record of instrument servicing maintained in a logbook and is it document controlled?	
☐	☐	Is appropriate corrective action taken if a surrogate is outside of acceptable limits?	
☐	☐	Are boiling chips kept to a minimum size to prevent volumetric distortion and minimize sample contamination potential? (only 1 boiling chip, the size of a grain of salt should be used)	
☐	☐	Are graduated cylinders utilized for measuring out sample volumes for water extractions?	

ATTACHMENT 5

LABORATORY AUDIT CHECKLIST

2.0 Organic Sample Preparation (continued)

Y	N	Item	Comment
		Are amber screw cap vials utilized for solvent extracts? (screw cap vials prevent solvent losses associated with crimp top vials)	
		If the volume of solvent extracts is reduced utilizing gas blowdown systems, do the gas lines contain molecular sieves to remove contaminants from the gas supply?	
		Are surrogates added to samples utilizing class A pipets in a manner to ensure quantitative transfer of the surrogates?	
		Are various dry reagents such as florisil, sodium sulfate and glass wool or thimbles, preextracted prior to use?	
		Is florisil PR 60/100 mesh utilized and is it stored in a dark glass bottle with foil lined cap and initially activated at 676 deg. C and at 130 ° C before use?	
		Is anhydrous sodium sulfate used which is dried at 400 ° C for 4 hours and stored at 130 ° before use?	
		Are only approved solvent lot numbers being utilized by the laboratory?	
		Is laboratory glassware rotated so that method blanks are truly representative of the effectiveness of routine glassware cleaning procedures? (dedicated glassware must not be used for QC samples) and only 1 blank can be extracted per batch.	
		When utilizing liquid/liquid continuous extractors is <u>all</u> the solvent remaining after extraction in both the extractor and the boiling flask concentrated down to the final volume?	
		Do the laboratory personnel indicate a familiarity with the requirements set forth in the corporate QA program?	

ATTACHMENT 5

LABORATORY AUDIT CHECKLIST

3.0 Gas Chromatography

Check off which of the following items are utilized in this area and complete the associated forms.

☐	☐	Refrigerators., freezers, ovens,	Complete Form A
☐	☐	Logbooks	Complete Form B
☐	☐	Laboratory Balances	Complete Form C
☐	☐	Reagents and Standards	Complete Form D
☐	☐	Safety Equipment	Complete Form E

Y	N	Item	Comment
☐	☐	Do the laboratory personnel indicate a familiarity with the requirements set forth in the laboratory QA program?	
☐	☐	Are SOPs available detailing the procedures used in this area?	Obtain a listing of all procedures performed in the area. Compare the list with available SOPs and estimate a percentage complete below. Percent Compliance _____
☐	☐	Are gas lines checked for leaks each time a tank is changed?	
☐	☐	Are instruments properly vented or are appropriate traps in place?	
☐	☐	Can the lab demonstrate the absence of volatile organics in the lab reagent water?	
☐	☐	Are SOPs available detailing the procedures used in this area?	
☐	☐	Are the solvent storage cabinets properly vented as appropriate to avoid possible laboratory contamination?	
☐	☐	Are operating manuals available to the analysts for all major equipment?	
☐	☐	Is there a record of instrument servicing maintained in a logbook and is it document controlled?	
☐	☐	Are volatile analysis being conducted using the appropriate packed or capillary columns as specified by the particular method?	
☐	☐	Is there a policy in place concerning carryover prevention in the event a high level sample precedes a relatively low level sample?	
☐	☐	Is quantitation correct for the method? (based upon the average RF of a calibration curve or the RF from a continuing calibration standard)	

ATTACHMENT 5

LABORATORY AUDIT CHECKLIST

3.0 Gas Chromatography (continued)

Y	N	Item	Comment
		Is the initial calibration conducted at five or three concentrations as appropriate for the method?	
		Are all reported results within the calibration range of the highest and lowest standard?	
		Are calibrations performed at the required frequency specified in the methods?	
		Is appropriate corrective action taken if a calibration is out of criteria?	
		Is a method blank run with every batch of samples?	
		Is there specified acceptance limits for the method blank?	
		Is there a policy stating that the laboratory does not "blank correct" sample data?	
		Is appropriate corrective action taken if a surrogate is outside of acceptance limits?	
		Is appropriate corrective action taken if internal standard area is outside of acceptance limits?	
		Is a run log maintained for all instrumentation?	
		Are GC identifications confirmed on a second dissimilar column?	
		Are PCB identifications performed by manual pattern recognition?	
		Are a sufficient number of peaks used to calculate a PCB result? (minimum of 3)	
		Are various extract cleanup techniques such as copper, florisil, alumina, sulfuric acid, used routinely?	
		Do gas lines contain molecular sieves or other filters to remove contaminants from the gas supply?	
		If 25 ml purge vessels are utilized for methods normally requiring a 5 ml purge, have MDL studies been performed to verify that required MDLs are achieved?	

ATTACHMENT 5

LABORATORY AUDIT CHECKLIST

4.0 Mass Spectrometry

Volatile GC/MS Area

Check off which of the following items are utilized in this area and complete the associated forms.

☐	Refrigerators., freezers, ovens,	Complete Form A
☐	Logbooks	Complete Form B
☐	Laboratory Balances	Complete Form C
☐	Reagents and Standards	Complete Form D
☐	Safety Equipment	Complete Form E

Y	N	Item	Comment
☐	☐	Do the laboratory personnel indicate a familiarity with the requirements set forth in the laboratory QA program?	
☐	☐	Are SOPs available detailing the procedures used in this area?	Obtain a listing of all procedures performed in the area. Compare the list with available SOPs and estimate a percentage complete below. Percent Compliance _____

Are the following QC samples processed with each analytical batch?

☐	☐	Method Blank	
☐	☐	QC Check Sample	
☐	☐	Matrix Spike	
☐	☐	Matrix Spike Duplicate	
☐	☐	Are gas lines checked for leaks each time a tank is changed?	
☐	☐	Are the instruments properly vented or are appropriate traps in place?	
☐	☐	Can the lab demonstrate the absence of volatile organics in the lab reagent water?	
☐	☐	Are the solvent storage cabinets properly vented as appropriate to avoid possible laboratory contamination?	
☐	☐	Is the laboratory utilizing the most recent version of the EPA/NIH mass spectral database? If not what version is being used?	
☐	☐	Are operating manuals available to the analysis for all major equipment?	
☐	☐	Is there a record of instrument servicing maintained in a logbook and is it document controlled?	
☐	☐	Are volatile analyses being conducted using the appropriate packed or capillary columns as specified by the particular method?	

ATTACHMENT 5

LABORATORY AUDIT CHECKLIST

	Is there a policy in place concerning carryover prevention in the event a high level sample precedes a relatively low level sample?	

ATTACHMENT 5

LABORATORY AUDIT CHECKLIST

4.0 Mass Spectrometry (continued)

Volatile GC/MS Area			
Y	N	Item	Comment
		Is the initial calibration conducted at five or three concentrations as appropriate for the method?	
		Is quantitation correct for the method? (based upon the average RF of a calibration curve or the RF from a continuing calibration standard)	
		Are calibrations and tuning performed according the required frequency specified in the methods?	
		Is appropriate corrective action taken if a calibration is out of criteria?	
		Is appropriate corrective action taken if a continuing calibration is out of criteria?	
		Is a method blank run with every batch of samples?	
		Is there specified acceptance limits for the method blank?	
		Is there a policy stating that the laboratory does not "blank correct" sample data?	
		Is the appropriate corrective action taken if a surrogate is outside of acceptance limits?	
		Is the appropriate corrective action taken if internal standard area is outside of acceptance limits?	
		Are library searches examined by the mass spectrometry expert before results are reported and is there documentation of this review?	
		Is a run log maintained for all instrumentation and is it documented controlled?	
		Is raw data stored in a secure manner under appropriate conditions to prevent loss of data?	
		Are the reported results within the calibration range of the highest and lowest standard?	
		Are appropriate dilutions performed?	
		Do gas lines contain molecular sieves or other filters to remove contaminants in gas supply?	
		If 25-ml purge vessels are utilized for methods normally requiring a 5-ml purge, have MDL studies been performed to verify that required MDLs are achieved?	

ATTACHMENT 5

LABORATORY AUDIT CHECKLIST

4.0 Mass Spectrometry (continued)

Semi-Volatile GC/MS Area		
Check off which of the following items are utilized in this area and complete the associated forms.		
	Refrigerators., freezers, ovens,	Complete Form A
	Logbooks	Complete Form B
	Laboratory Balances	Complete Form C
	Reagents and Standards	Complete Form D
	Safety Equipment	Complete Form E

Y	N	Item	Comment
		Do the laboratory personnel indicate a familiarity with the requirements set forth in the laboratory QA program?	
		Are SOPs available detailing the procedures used in this area?	Obtain a listing of all procedures performed in the area. Compare the list with available SOPs and estimate a percentage complete below. Percent Compliance _____
		Are gas lines checked for leaks each time a tank is changed?	
		Are the instruments properly vented or are appropriate traps in place?	
		Can the lab demonstrate the absence of volatile organics in the lab reagent water?	
		Are the solvent storage cabinets properly vented as appropriate to avoid possible laboratory contamination?	
		Is the laboratory utilizing the most recent version of the EPA/NIH mass spectral database? If not what version is being used?	
		Are operating manuals available to the analysis for all major equipment?	
		Is there a record of instrument servicing maintained in a logbook and is it document controlled?	
		Are volatile analyses being conducted using the appropriate packed or capillary columns as specified by the particular method?	
		Is there a policy in place concerning carryover prevention in the event a high level sample precedes a relatively low-level sample?	
		Is the initial calibration conducted at five or three concentrations as appropriate for the method?	

ATTACHMENT 5

LABORATORY AUDIT CHECKLIST

4.0 Mass Spectrometry (continued)

Semi-Volatile GC/MS Area			
Y	N	Item	Comment
		Is quantitation correct for the method? (based upon the average RF of a calibration curve or the RF from a continuing calibration standard)	
		Are calibrations and tuning performed according the required frequency specified in the methods?	
		Is appropriate corrective action taken if a calibration is out of criteria?	
		Is appropriate corrective action taken if a continuing calibration is out of criteria?	
		Is a method blank run with every batch of samples?	
		Is there specified acceptance limits for the method blank?	
		Is there a policy stating that the laboratory does not "blank correct" sample data?	
		Is the appropriate corrective action taken if a surrogate is outside of acceptance limits?	
		Is the appropriate corrective action taken if internal standard area is outside of acceptance limits?	
		Are library searches examined by the mass spectrometry expert before results are reported and is there documentation of this review?	
		Is a run log maintained for all instrumentation and is it documented controlled?	
		Is raw data stored in a secure manner under appropriate conditions to prevent loss of data?	
		Are the reported results within the calibration range of the highest and lowest standard?	
		Are appropriate dilutions performed?	
		Do gas lines contain molecular sieves or other filters to remove contaminants in gas supply?	

ATTACHMENT 5

LABORATORY AUDIT CHECKLIST

5.0 Metals Preparation

Check off which of the following items are utilized in this area and complete the associated forms.		
☐	Refrigerators, freezers, ovens,	Complete Form A
☐	Logbooks	Complete Form B
☐	Laboratory Balances	Complete Form C
☐	Reagents and Standards	Complete Form D
☐	Safety Equipment	Complete Form E

Y	N	Item	Comment
☐	☐	Is the storage area for acids properly ventilated?	
☐	☐	Do the laboratory personnel indicate a familiarity with the requirements set forth in the laboratory QA program?	
Are the following QC samples processed with each analytical batch?			
☐	☐	Method Blank	
☐	☐	QC Check Sample	
☐	☐	Matrix Spike	
☐	☐	Matrix Spike Duplicate or Matrix Duplicate	
☐	☐	Are SOPs available detailing the procedures used in this area?	Obtain a listing of all procedures performed in the area. Compare the list with available SOPs and estimate a percentage complete below. Percent Compliance _____
☐	☐	Is there a SOP for glassware washing and is it posted in the washing area?	
☐	☐	Are high purity trace metal grade acids utilized for preservation and digestion of samples for metals?	
☐	☐	Is glassware for metals analysis acid washed?	
☐	☐	If automatic dispensers are utilized for acids, are they properly maintained?	
☐	☐	Does the lab ensure that powder free gloves are used to avoid possible zinc contamination of samples?	
☐	☐	Are operating manuals available to the analysts for all major equipment?	

ATTACHMENT 5

LABORATORY AUDIT CHECKLIST

5.0 Metals Preparation (continued)

Y	N	Item	Comment
		Is there a record of instrument servicing maintained in a logbook and is it document controlled?	
		Is a digestion log maintained for all samples and is it document controlled?	
		Is an acid digestion performed for drinking water samples requiring total metals?	
		Are samples for dissolved metals digested after filtration?	EPA required, except for DW where turbidity is below a certain level.

ATTACHMENT 5

LABORATORY AUDIT CHECKLIST

6.0 Metals Analysis

Check off which of the following items are utilized in this area and complete the associated forms.

☐	Refrigerators., freezers, ovens,	Complete Form A
☐	Logbooks	Complete Form B
☐	Laboratory Balances	Complete Form C
☐	Reagents and Standards	Complete Form D
☐	Safety Equipment	Complete Form E

Y	N	Item	Comment
☐	☐	Do the laboratory personnel indicate a familiarity with the requirements set forth in the laboratory QA program?	
☐	☐	Are SOPs available detailing the procedures used in this area?	Obtain a listing of all procedures performed in the area. Compare the list with available SOPs and estimate a percentage complete below. Percent Compliance _____
☐	☐	Are gas lines checked for leaks each time a tank is changed?	
☐	☐	Are vents above AA units functioning properly (200 ft ³ /min.)?	
☐	☐	Are SOPs available detailing the procedures used in this area?	
☐	☐	Are operating manuals available to the analysis for all major equipment?	
☐	☐	Is there a record of instrument servicing maintained in a logbook and is it document controlled?	
☐	☐	Is there a policy in place concerning carryover prevention in the event a high level sample precedes a relatively low-level sample?	
☐	☐	Are calibrations performed according the required frequency specified in the methods?	
☐	☐	Is appropriate corrective action taken if a calibration is out of criteria?	
☐	☐	Are continuing calibrations run at an adequate frequency?	

ATTACHMENT 5

LABORATORY AUDIT CHECKLIST

6.0 Metals Analysis (continued)

Y	N	Item	Comment
		Is appropriate corrective action taken if a continuing calibration is out of criteria?	
		Is a method blank run with every batch of samples?	
		Is there specified acceptance limits for the method blank?	
		Is there a policy stating that the laboratory does not "blank correct" sample data?	
		Is a run log maintained for all instrumentation and is it documented controlled?	
		Are post-digestion spikes performed for graphic furnace analyses?	
		Is there a corrective action when PDS fail? Is it to flag results or perform standard additions?	
		Are ICP interference standards run at the required frequency specified in the method?	
		Are serial dilutions performed?	
		Are check standards run at a frequency of 10% for graphite furnace to monitor when the furnace should be changed?	
		For mercury analysis, is the mercury vapor safely removed from the lab atmosphere?	
		Are all reported results within the calibration range of the highest and lowest standard?	

ATTACHMENT 5

LABORATORY AUDIT CHECKLIST

7.0 Wet Chemistry

Check off which of the following items are utilized in this area and complete the associated forms.		
☐	Refrigerators., freezers, ovens,	Complete Form A
☐	Logbooks	Complete Form B
☐	Laboratory Balances	Complete Form C
☐	Reagents and Standards	Complete Form D
☐	Safety Equipment	Complete Form E

Y	N	Item	Comment
		Are SOPs available detailing the procedures used in this area?	Obtain a listing of all procedures performed in the area. Compare the list with available SOPs and estimate a percentage complete below. Percent Compliance _____
Are the following QC samples processed with each analytical batch?			
		Method Blank	
		QC Check Sample	
		Matrix Spike	
		Matrix Spike Duplicate or Matrix Duplicate	
		If the lab utilizes dessicators, do they appear to be properly maintained?	
		Is there a SOP for glassware washing and is it posted in the washing area?	
		Are operating manuals available to the analysts for all major equipment?	
		Is there a record of instrument servicing maintained in a logbook and is it document controlled?	
		Is there a policy in place concerning carryover prevention in the event a high level sample precedes a relatively low-level sample?	
		Are calibrations performed according the required frequency specified in the methods?	
		Is appropriate corrective action taken if a calibration is out of criteria?	

ATTACHMENT 5

LABORATORY AUDIT CHECKLIST

7.0 Wet Chemistry (continued)

Y	N	Item	Comment
		Is a method blank run with every batch of samples?	
		Is there specified acceptance limits for the method banks?	
		Is there a policy stating that the laboratory does not "blank correct" sample data?	
		Is a dedicated analysis log maintained for all parameters determined and are they document controlled?	
		Is the pH meter checked daily, or before use, by setting the meter to pH 7.0, then measuring pH at 4 and 10?	
		Is the conductivity meter calibrated daily against a 0.001MKCl solution and the data recorded in a logbook?	
		Is the turbidometer calibrated daily, or before use, with 40 NTU formazine standard and the data recorded in a logbook?	
		Is the dissolved oxygen meter calibrated weekly against the Winkler method and data recorded in a logbook?	
		Are all reported results within the calibration range of the highest and lowest standard?	

ATTACHMENT 5

LABORATORY AUDIT CHECKLIST

8.0 Report Generation

Check off which of the following items are utilized in this area and complete the associated forms.			
☐	☐	Logbooks	Complete Form B
☐	☐	Safety Equipment	Complete Form E

Y	N	Item	Comment
☐	☐	Do the laboratory personnel indicate a familiarity with the requirements set forth in the laboratory QA program?	
☐	☐	Are SOPs available detailing the procedures used in this area?	Obtain a listing of all procedures performed in the area. Compare the list with available SOPs and estimate a percentage complete below. Percent Compliance _____
☐	☐	Is there a document describing in detail the contents and order of deliverables provided with various types of reporting levels or packages?	
☐	☐	Do all reports contain a cover letter and case narrative?	
☐	☐	Is chain of custody provided with all reports?	
☐	☐		
☐	☐		
☐	☐		

ATTACHMENT 5

LABORATORY AUDIT CHECKLIST

9.0 Computer Systems

Check off which of the following items are utilized in this area and complete the associated forms.

		Logbooks	Complete Form B
		Safety Equipment	Complete Form E
Y	N	Item	Comment
		Do the laboratory personnel indicate a familiarity with the requirements set forth in the laboratory QA program?	
		Are SOPs available detailing the procedures used in this area?	Obtain a listing of all procedures performed in the area. Compare the list with available SOPs and estimate a percentage complete below. Percent Compliance _____
		Are computer programs validated before use?	
		Are operating manuals available to the computer specialists for all computer equipment?	
		Is there a record of computer servicing maintained in a logbook and is it document controlled?	
		Is the computer system backed up at a regular frequency and if so, what is the frequency? Backup copy maintained off Site?	
		Is the system protected from unauthorized access utilizing a password system?	
		Are backup power supplied utilized to prevent loss of information in the event of a power failure?	
		Is the system protected from a lightning strike?	
		If an in-house information management system is utilized, are instructions available to the users?	
		Is there a formal training program for new users?	
		Is there any mechanism to prevent computer clocks from being altered to reflect incorrect dates and times?	
		If such alteration would occur, is there a mechanism to detect it in a <u>very</u> timely fashion?	
		Are computer specialists at the facility familiar with the EPA computer requirements set forth in the GALP manual (good automated lab practices)?	

ATTACHMENT 5

LABORATORY AUDIT CHECKLIST

Form A - Laboratory Temperature Monitoring Checklist

Identify the Laboratory Area under Review:			
Identify the number of units in this laboratory area:			Refrigerators: Freezers: Ovens:
Y	N	Item	Comment
		Do the laboratory personnel indicate a familiarity with the requirements set forth in the laboratory SOP for temperature monitoring?	
		Are temperatures of all lab temperature units such as refrigerators, ovens, freezers and incubators monitored daily and documented in a logbook?	
		Are all units individually identified with a unique identification number on the outside of the unit?	
		Have thermometers been calibrated against NIST within the last year and documented?	
		Are temperature excursion noted and appropriate action taken?	
		Does all laboratory equipment contain thermometers, which are uniquely identified?	
		Are all thermometers immersed in appropriate media?	
		Is the laboratory aware of whether the temperature has been checked routinely?	

ATTACHMENT 5

LABORATORY AUDIT CHECKLIST

Form B - Laboratory Notebook Checklist

Identify the Laboratory Area under Review:			
Identify the Number of Notebooks in this Area:			
Y	N	Item	Comment
		Do the logbooks comply with GLP standards for logbooks?	
		Are the contents of the notebook clearly identified?	
		Are lab notebooks maintained in black ink?	
		Are notebooks permanently bound and contain preprinted and consecutively numbered pages?	
		Are lab notebooks neat, orderly and legible?	
		Are entries noting anomalies routinely recorded?	
		Are mistakes in notebooks crossed out with one line signed and dated?	
		Do all notebooks have a unique document control number?	
		Is there evidence of secondary review of the notebook?	
		Are unused portions of pages struck out with a line or Z'd out?	

ATTACHMENT 5

LABORATORY AUDIT CHECKLIST

Form C – Laboratory Balance Checklist

Identify the Laboratory Area under Review:			
Identify the Number of Balances in this Area:			
Y	N	Item	Comment
		Are all balances being calibrated at the correct frequency?	
		Have the calibrations been conducted at the correct level?	
		Are the acceptance limits presented in the logbook?	
		Are balances and S weights maintained in a clean environment?	
		Does each balance have a unique identification number?	
		Has each balance been serviced in the past year and is a sticker affixed to the balance?	

ATTACHMENT 5

LABORATORY AUDIT CHECKLIST

Form D - Laboratory Reagents and Standards Checklist

Identify the Laboratory Area under Review:			
Y	N	Item	Comment
		Is there a SOP describing how reagents and standards are prepared and stored?	
		Is there a mechanism for verifying a prepared standard or purchased commercial standard prior to its use in the lab?	
		Is a standards notebook maintained for all areas, which describes the preparation and standardization of standard solutions and is it document controlled?	
		Are stock standards traceable to their source?	
		Are standards and reagents properly labeled with received date, concentration, date prepared, preparer's initials and expiration date?	
		Are standards stored separately from samples or sample extracts?	
		Are all sources of reagent water checked daily?	
		Does water meet the conductance requirements outlined in the SOP and is it documented?	
		Are the water quality records document controlled?	
		Are reagent grade or higher-grade chemicals used to prepare standards?	
		Is there evidence that all reagents and standards are within their expiration dates?	
		Are chemicals being used in analyses tested to ensure they contain no contaminants, which may interfere with the analyses, prior to use?	

ATTACHMENT 5

LABORATORY AUDIT CHECKLIST

Form E - Laboratory Safety Checklist

Identify the Laboratory Area under Review:			
Y	N	Item	Comment
		Is there sufficient safety equipment in this area such as fume hoods, shower, eyewash, first aid kit, etc.?	
		Is there an emergency spill kit available in the vicinity?	
		Are lab fume hoods checked monthly for proper airflow and is it documented?	
		Is there a cutoff level for determining airflow (100fpm)?	
		Is the interior of fume hoods maintained in a clutter free manner?	
		Are safety glasses worn in the laboratory areas?	
		Are appropriate gloves worn suitable for the reagents used in the laboratory?	
		Are the storage areas and benchtops maintained in an orderly manner such that safety hazards are avoided?	
		Have fire extinguishers, safety showers, eyewashes been checked for proper operation?	
		Are self-contained breathing apparatus available for use in emergency situations?	
		Is all standards preparation performed in a fume hood?	
		Are material safety data sheets (MSDS) accessible and up to date?	
		Are all instruments properly vented?	
		Have all personnel participated in formal safety training?	