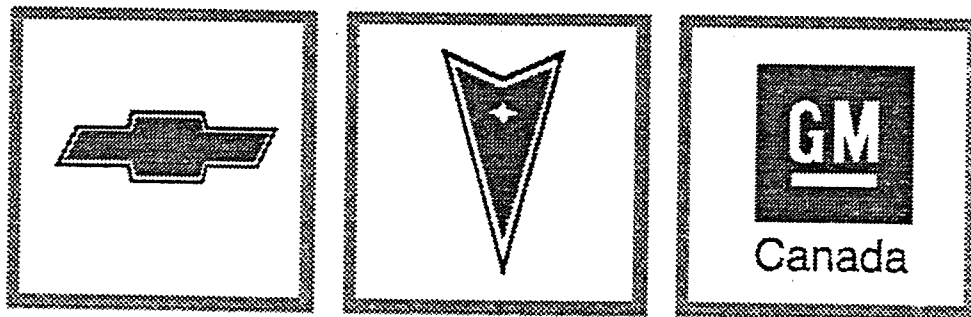


# TCE Remediation Monitoring Program

for



General Motors Corporation  
CPC Group

Grand Rapids, Michigan

March, 1989

21099

**EDI Engineering & Science**

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## **1.0 GROUND WATER MONITORING**

### **1.1 INTRODUCTION**

A plume of dissolved trichloroethylene (TCE) extends to the north property line of the General Motors Corporation/CPC Group Metal Fabrication Plant in Wyoming, Michigan. The soils below the floor also have residuals of TCE. In previous hydrogeological investigations by EDI the source was identified in the plant at Column T-27 where a parts cleaner leaked TCE. This equipment was removed from the plant in 1979.

A series of monitoring wells have been installed; and a monitoring program has been initiated. In addition, a monitoring well at Column X-10, and a purge well near 86-3 will be installed during 1989. Monitoring of these new wells will begin when they are completed in the summer of 1989.

The individual components of the ground water monitoring program are described in this chapter. The location and construction of the sampling sites which form this ground water monitoring system are described in Section 1.2. These sites include monitoring wells screened in an unconfined aquifer. The constituents which will be monitored at each of these sampling sites is presented in Section 1.3 and the frequency of this monitoring is described in Section 1.4. Section 1.5 through 1.8 present a detailed discussion of the sampling, analysis and documentation procedures which will be used in this monitoring program; Section 1.9 pertains to the reporting. Surface water sampling in Cole Drain is described in Section 2.

### **1.2 GROUND WATER MONITORING SITES**

#### **1.2.1 Monitoring Well Locations**

To evaluate the progress of the clean up, the monitoring program includes monitoring the concentration of TCE in the groundwater surrounding the source area and the plume. In order to determine the direction of ground water flow and the effect of the purge wells, the elevation of the water table will be measured in 22 monitoring wells and 2 purge wells. The wells which will be used to monitor these tasks are listed in Table 1; their locations are shown on Figure 1; and the well logs are included in Appendix 1.

North of the Metal Fabrication plant property, ground water flows from the southeast to the northwest in the area east of Cole Drain and from the southwest to the northeast in the area west of Cole Drain (Figure 2). As determined from previous sampling and water

level data, the ground water eventually discharges into Cole Drain. Monitor well 87-2 is located upgradient and will be used to collect samples which are representative of the ground water before it flows under the source area. The monitoring well to be installed at Column X-10 (monitoring to begin in the summer of 1989) will be placed in the zone of highest TCE concentration and will provide advance notice of the trend to be expected in the North Lot Line Purge Well.

### 1.2.2 Monitoring Well Construction

Six monitoring wells 85-1, 85-2, 85-3, 85-5B, 85-6, 85-7 were installed in November and December of 1985. The wells were drilled using hollow stem augers; all of these wells were completed with 2-inch diameter galvanized steel casings attached to 2-inch diameter, #10 slot, wire-wrapped stainless steel screens. The screens were partially submerged below the water table so that floatable hydrocarbons could be detected and the thickness measured. The wells were backfilled with natural soils and bentonite; and cement grout was placed around the wells at the surface.

Three additional monitoring wells were installed in April and May of 1986. Monitoring wells 86-1 and 86-2 were installed using flights of hollow stem augers; 86-3 required a combination of hollow stem augers and mud rotary drilling methods. The well casing for 86-1 is a 2-inch diameter galvanized casing with a 2-inch diameter, #10 slot, 10-foot long stainless steel screen which was positioned so that it intercepted the water table. Wells 86-2 and 86-3 are also 2-inch diameter casings with 2-inch diameter, #10 slot, 5-foot long stainless steel screens. Monitoring well 86-2 was converted to a purge well in 1989.

Monitoring wells 87-1, 87-2, 87-4, 87-5, 87-8 and 87-9 were installed in January 1987 using flights of hollow stem augers. Wells 87-1, 87-2, and 87-4 are constructed of 2-inch diameter galvanized casings and 2-inch diameter, #7 slot, 3-foot stainless steel screens. Each of these wells has an upper section with 10 feet of stainless steel screen in the soils above the water table with a 1-foot connector in the middle. At each well the formation was allowed to collapse around the screens and the borings were backfilled with natural soils to the surface. The wells were completed with flush-mount caps cemented into the floor of the plant, level with the cement and below the wood block. Well 87-5 is constructed of 4-inch diameter galvanized casing with a 4-inch diameter, #10 slot, stainless steel screen. This screen has two sections with a 1-foot connector in the middle. The upper section, 10 feet in length, is located in the soils above the water table and 10 to 20 feet below the floor level. The lower 10 feet of the screen is set near

the bottom of the aquifer. The boring was backfilled with natural soils to the surface. Well 87-5 was completed with a standard cap below grade, inside a 12-inch diameter steel ring and cover, set level with the concrete floor. Monitoring wells 87-8 and 87-9 are constructed of 2-inch diameter galvanized casings and 2-inch diameter, #7 slot, 3-foot long stainless steel screens. At these wells the formation was allowed to collapse around the screens, and the boring was backfilled to the surface with natural soils and bentonite.

Monitoring wells 87-10, 87-11 and 87-13 were installed in October 1987 and monitoring wells 88-1, 88-2, 88-3 and 88-4 were installed in March and April 1988. All of these wells, except 87-10, were drilled using flights of hollow stem augers. 87-10 was drilled using hollow stem augers and mud rotary drilling techniques. The wells are constructed with 2-inch diameter galvanized steel casings attached to 3-foot long, 2-inch diameter, #7 slot wire-wrapped stainless steel screens. Monitoring wells 87-10, 87-11 and 87-13 were set within the most contaminated interval at 29 to 32, 30 to 33 and 40 to 43 feet respectively; wells 88-1, 88-2, 88-3 and 88-4 were set at 27 to 30, 26 to 29, 27 to 30 and 18 to 21 feet below the ground surface. The monitoring wells were backfilled with natural soils around the screen and a mixture of natural soils and bentonite up to or near the surface.

Unless otherwise noted, all of the monitoring wells described above have flush mount caps placed at the surface, internally locking caps and cement around the ground level portion of the caps.

### **1.3 CONSTITUENTS TO BE MONITORED**

The unconfined aquifer under the Metal Fabrication Plant will be monitored for trichloroethylene and the cis and trans isomers of 1,2-dichloroethylene (Table 2, set A) in the monitoring wells which are indicated with an "A" indicated in Table 1. Ground water samples from six of these wells, indicated with a "B" in Table 1, will also be analyzed for 1,1-dichloroethylene and vinyl chloride (Table 2, set B) during the fourth quarter. Beginning in the summer of 1989, the discharge from the purge well to be installed in the North Lot Line (PW disch) will be monitored for trichloroethylene and the cis and trans isomers of 1,2-dichloroethylene (Table 2, set A) and during the fourth quarter the discharge will also be monitored for 1,1-dichloroethylene and vinyl chloride (Table 2, set B). In addition, purge well 86-2 and MW at X-10 will be analyzed for trichloroethylene (Table 2, set C).

## 1.4 SAMPLING FREQUENCY

Ground water will be sampled monthly in triplicate (three containers per site per sampling event) from purge well 86-2, from the monitoring well to be installed at Column X-10, and from the new North Lot Purge Well (monitoring to begin Summer 1989). A treated discharge sample from purge well 86-2 will also be sampled monthly. One of the three samples from each of these wells will be analyzed as discussed in Section 1-3. Monitoring wells listed in Table 1 and indicated with an "A" will be sampled in triplicate on a quarterly basis, but only one of the three samples will be analyzed. On a monthly basis, water levels will be measured in monitoring and purge wells labelled with an "X"; and the presence and thickness of oil will be monitored in wells labelled with an "O", as indicated in Table 1.

## 1.5 SAMPLE COLLECTION

The procedures used to sample water can control the quality of the data produced in a monitoring program. Muska and others, (1986) performed a field evaluation of ground water monitoring devices for volatile organic compounds and showed that "by using careful and reproducible procedures, overall sampling variability is low regardless of (the) sampling device (used)."

Following the sample collection procedures described in this section will generate samples and data which are representative of the actual conditions which exist on the site at the time of the sampling. These procedures are also designed to supply sufficient documentation of the sample collection procedures so that future uses of the data can reevaluate the sample collection procedures. The method used to collect the water samples and the field data will be discussed first, followed by the methods used to document the actual methods used in the field.

### 1.5.1 Measurement of Static Water Level

Static water levels will be measured in monitoring wells using an electric tape or a chalked steel tape. The distance from the top of the casing (T.O.C) to the static water level will be measured to within 0.01 foot. The elevation of the static water level is calculated by subtracting the distance from the T.O.C. to the static water level from the elevation of the T.O.C. The elevations of the top of the casing on the monitoring wells are listed in Table 3. Well 86-2 was converted to a purge well as discussed in Section 1.2. Water levels in the purge well will be measured with an air flow tube, which was

installed adjacent to the pump suction line in the well casing. The tube extends below the water table. Air will be injected into the tube and a gauge attached to the tube will indicate the static water level to within 0.1 foot. Water levels will be measured prior to purging any well to assure they are not affected by the withdrawal of ground water during the purging activity. The device used to measure the water levels will be washed with distilled water after each measurement. However, in the wells which have floating oil product, the device will be washed withalconox and rinsed with distilled water after each measurement.

### 1.5.2 Sampling Equipment

Wells are sampled using Teflon or stainless steel bailers with polypropylene rope. Clear acrylic bailers will be used in wells in which the thickness of floating oil product will be measured.

### 1.5.3 Well Evacuation

All monitoring wells will be purged of three casing volumes prior to the collection of a ground water sample. The purge volume will be calculated using the following equation:

$$V = 3 \pi r^2 [(well\ depth\ in\ feet)-(depth\ to\ water\ in\ feet)],$$

where  $V$  = purge volume

$$\pi = 3.1416$$

$r$  = radius of the well

For a 2-inch diameter well this simplifies to:

$$V(\text{gal}) = 0.49[(well\ depth\ in\ feet)-(depth\ to\ water\ in\ feet)]$$

and for a 4-inch diameter well this simplifies to

$$V(\text{gal}) = 1.96[(well\ depth\ in\ feet)-(depth\ to\ water\ in\ feet)]$$

The bailers are cleaned withalconox and distilled water between wells.

### 1.5.4 Sample Withdrawal

Ground water will be withdrawn from each of the monitoring wells using Teflon or

stainless steel bailers as described in Section 1.5.2. The bailers will be gently lowered into the water; and samples will be collected by gently pouring the water into glass vials, filled just to overflowing, ensuring that no air bubbles pass through the sample as the vial is being filled. Each vial will be sealed with a clean Teflon-lined septa and cap so that no headspace or entrapped air bubbles are present in the sample. Ground water will also be collected from purge well 86-2. The material being sampled will be allowed to run for several minutes to ensure that the sample is representative of the material to be analyzed. The samples will be poured and sealed in glass vials as described above. Sample preservation and shipment are discussed in Section 1.6.

#### 1.5.5 Sample Blanks

During each quarterly and monthly sampling event, one trip blank and one field blank will be analyzed for each parameter. When the sample bottles are prepared prior to shipment to the field, two glass vials will be filled with deionized water, and labeled "trip blank". The trip blank will be transported to the field and sent to the laboratory for analysis along with the other sample bottles to evaluate possible sample contamination from the sample bottles, shipping methods, or laboratory analyses. One field blank will be analyzed to ensure that sampling methods have not affected the quality of the samples and that equipment has been adequately cleaned. The field blank will be filled with deionized water in the field, labeled "field blank" and sent to the laboratory for analysis along with the other sample bottles.

In the laboratory, the trip and field blanks will be analyzed for the same parameters as the samples. The presence of any contaminant in the trip blank will be noted and attributed to sample contamination; and the presence of any contaminant in the field blank will be noted and attributed to sampling methods or equipment. This data will not be used to correct the concentrations in other samples.

#### 1.5.6 Documentation of Sample Collection

The sampling equipment and procedures described in the previous sections are designed to generate data which is representative of the ground water under the Metal Fabrication Plant and the surrounding area. To properly use this data to evaluate the progress of the clean-up of the ground water and soils, the actual procedures used in the field to collect the samples must be documented. As a result, future users of the data can review and reevaluate any possible bias introduced by these procedures. The documentation procedures which will be used are described below.

Customized field data entry forms will be used to assure that the sampling procedures are thoroughly documented. The initial field data entry forms are included in Appendix 2. These forms are designed both to provide thorough documentation and to aid in the efficient performance of the sampling event. For instance, space is provided to calculate purge volumes from the static water levels, providing both documentation and a convenient worksheet in the field.

It is impossible to include all of the modifications in a monitoring program necessary for any type of field condition; likewise, field samplers may be confronted with field conditions which make it impossible to follow the designed sample collection procedures. If this occurs, the field sampler will note these changes on the field data entry form so that the forms always represent the actual procedures used during the sample collection. When the field sampler completes a sampling event she/he will have filled in all of the information on the field data entry forms and will have noted any deviations from the sample procedures. The field sampler will then sign the forms; and the forms will be kept as documentation of the actual sampling procedures used.

#### **1.6 SAMPLE PRESERVATION, SHIPMENT AND ANALYTICAL PROCEDURES**

Complete and unequivocal preservation of samples is practically impossible. Preservation techniques are used to retard the chemical and biological changes that may take place after a sample is taken from its parent source. Samples collected in this detection monitoring program will be placed in coolers immediately upon collection and then transported to the appropriate analytical laboratory where they will be stored at 4°C until analysis. Ground water samples will be preserved according to the Friday, October 26, 1984 Federal Register, Volume 49, #209, 43260. Samples for the analysis of volatile organic compounds will be collected in a triplicate, 40-ml glass vials and sealed with Teflon-lined caps. No preservative is required, but samples will be kept cool at 4°C. All volatiles will be analyzed within 14 days (the maximum holding time,) using method 624 as referenced in the July 1982 USEPA-600/4-82-057.

#### **1.7 CHAIN OF CUSTODY**

A chain-of-custody procedure will be established to document the possession and handling of individual samples from the field collection activities through laboratory analysis. This program will include sample identification labels, field data entry forms which contain field sampling observations and data (see Section 1.5.6), chain-of-custody records which include documentation of sample receiving and transportation, and

laboratory logbooks which include information about the analyses of the sample in the laboratory. The chain-of-custody form used at EDI Engineering & Science is in Appendix 3. The chain-of-custody program used by the ASI laboratory performing a portion of the analyses will be at least as thorough.

## **1.8 QA/QC PROCEDURES**

The analytical procedures which will be used are outlined above. These analytical procedures include the method detection limits which will be used in reporting the data (Table 2).

The quality assurance/quality control program for the laboratory at EDI Engineering and Science is presented in Appendix 3. The quality assurance/quality control program for the ASI laboratory which performs a portion of the analyses will be at least as thorough.

## **1.9 REPORTING**

EDI will submit quarterly reports to the CPC Group Metal Fabrication Plant, 45 days after the quarterly sampling event, for submittal to the Michigan Department of Natural Resources (MDNR) 60 days after the quarterly sampling event. These reports will include a summary of the analyses of the constituents listed in Table 2, a contour map of the water elevations from locations listed in Table 1 and a brief interpretation of the data. In addition, the water quality data will be summarized and the progress of the monitoring plan evaluated on an annual basis.

## **1.10 REFERENCES FOR SECTION 1**

Federal Register, October 26,; 1984. Vol. 49, #209, p. 43260.

Muska, C.F., W.P. Colven, V.D. Jones, J.T. Scogin, B.B Looney, and V. Price, Jr., 1986. "Field Evaluation of Ground Water Sampling Devices for Volatile Organic Compounds" in *Proceedings of the Sixth National Symposium and Exposition on Aquifer Restoration and Ground Water Monitoring*. National Water Well Association, Dublin, Ohio. p. 235-246.

USEPA-600/4-82-057, July 1982. "Methods for Organic Chemical Analysis of Municipal and Industrial Wastewater".

## **2.0 SURFACE WATER MONITORING (COLE DRAIN)**

### **2.1 SURFACE WATER SAMPLING LOCATIONS**

In addition to thoroughly monitoring the ground water, the monitoring program activities will include surface water sampling in Cole Drain from three locations along the area being monitored. Samples of the surface water will be collected at the sampling points listed in Table 1 and shown on Figure 1.

### **2.2 SURFACE WATER SAMPLING PLAN**

The surface water sampling plan is summarized in Table 1. Surface samples from Cole Drain will be collected quarterly in triplicate and one of the three samples from each site will be analyzed for parameters listed in Table 2, set A.

Currently, Cole Drain does not dry up during any time of the year. If conditions change so that Cole Drain does dry up during sampling periods, samples will be collected after a 24-hour, 0.5 inch rainfall.

### **2.3 SURFACE WATER SAMPLE COLLECTION**

#### **2.3.1 Measurement of Water Level**

The water level in Cole Drain will be measured monthly using an electric tape or a chalked steel tape. The distance from the top of the culvert to the water will be measured to within 0.01 foot. The elevation of the water level is calculated by subtracting the distance from the top of the culvert to the water level in Cole Drain from the elevation of the top of the culvert. The elevations of the fixed datum at each Cole Drain Location are listed in Table 3.

#### **2.3.2 Sample Withdrawal**

Grab samples will be collected at each of the surface water sampling sites listed in Table 1. The sampling bottles will be used to collect the surface water samples, so no sampling equipment or decontamination procedures are required.

#### **2.3.3 Sample Preservation, Shipment and Analysis**

Sample preservation, shipment and analysis procedures are described in Section 1.6 and 1.8. The chain-of-custody program which will be followed is described in Section 1.7.

## **2.4 REPORTING**

The data from the surface water monitoring will be reported to the MDNR as a part of the quarterly reports discussed in section 1.9.

## **3.0 SOIL VAPOR RECOVERY MONITORING**

### **3.1 SOIL VAPOR MONITORING LOCATIONS**

A soil vapor extraction system, with carbon adsorption treatment of the effluent, has been installed at monitoring well 87-3 at Column T-26 (Figure 1) to remove TCE from the soil. An air flow reading will also be taken each month to verify that pressure in the vacuum well remains steady.

Furthermore, pressure will be monitored at four additional sites near 87-3 to verify that the vapor extraction system is working effectively. The probe locations are shown on Figure 4.

Well 87-3 was installed in January 1987 and is constructed of 4 inch diameter galvanized casing and 20-foot long, #10 slot stainless steel screen. The boring was backfilled with natural soils. The well was completed with a standard cap below grade inside a 12-inch diameter steel ring and cover, set level with the concrete floor. A schematic diagram of the system is shown in Figure 3; and the well log is included in Appendix 1.

The pressure monitoring probes P1, P3, P4, and P5, which are located near 87-3, are 1/2-inch diameter, black carbon pipes with 3/32-inch diameter perforations on the lower 8 inches of the pipe. The probe, which has a tapered point, was driven into the soils; and the top of the probe was sealed into the floor. P1 is 10-feet long and P3, P4 and P5 are each 15 feet long. Pressure gauges were installed at the top of each probe and monitoring began in April of 1989. The pressure readings at the soil probes will enable EDI to verify that flow soil vapor is toward well 87-3.

### **3.2 SOIL VAPOR RECOVERY SAMPLING PLAN**

The soil vapor recovery sampling plan is summarized in Table 1. Air samples will be collected monthly from 87-3, beginning April 1989, and will be analyzed for TCE as listed in Table 2, set D. In addition, pressure readings and the air flow rate will be recorded each month at the locations discussed above.

### **3.3 SAMPLE WITHDRAWAL**

Air samples will be collected from 87-3 at two different sample ports. The soil vapor will be collected at the influent sample port and the treated discharge will be collected at the effluent sample port. The cap on the air sample tube will be removed just prior to sampling. Calibrated air flow is drawn from the sample port into sorbent air sample tubes containing a charcoal coconut base.

### **3.4 SAMPLE PRESERVATION AND SHIPMENT**

Air samples collected in this monitoring program will be kept at room temperature after collection. The samples will then be transported to the appropriate analytical laboratory as soon as possible where they will also be stored at 4°C until analysis. The charcoal tubes will be analyzed within 14 days.

**TABLES**

**TABLE 1**  
**MONITORING PROGRAM**  
**WELLS AND SAMPLING SITES**

Monitoring Sites	Monthly	Quarterly	Additional Annual	Monthly Water Level	Monthly Pressure Reading	Monthly Floating Oil
<b>Monitoring Wells</b>						
85-1				X		
85-2				X		
85-3				X		0
85-5B				X		0
85-6				X		0
85-7		A		X		
86-1				X		0
86-3		A	B	X		
87-1		A		X		
87-2		A		X		
87-4		A		X		
87-5		A		X		
87-8		A	B	X		
87-9		A	B	X		
87-10		A	B	X		
87-11		A	B	X		
87-13		A	B	X		
88-1				X		
88-2				X		
88-3				X		
88-4				X		
MW at X-10	C			X		
<b>Vapor Well</b>						
87-3/Soil vapor	D				X	
87-3/Treated vapor	D					
P1					X	
P3					X	
P4					X	
P5					X	
<b>Purge Wells</b>						
86-2/Pumped	C			X		
86-2/Treated	C					
PW disch	A		B	X		
<b>Surface Water</b>						
C-1		A		X		
C-2		A		X		
C-3		A				
C-4				X		

**TABLE 2**  
**MONITORING PARAMETERS**

	Parameter	Expected Detection Limit*(ug/l)
A	1,2-Dichloroethylene**	2.0
	Trichloroethylene	2.0
B	1,1-Dichloroethylene	2.0
	Vinyl Chloride	10.0
C	Trichloroethylene (Water Analysis)	2.0
D	Trichloroethylene (Air Analysis)	
	charcoal tube	
	soil vapor	500
	treated vapor	1

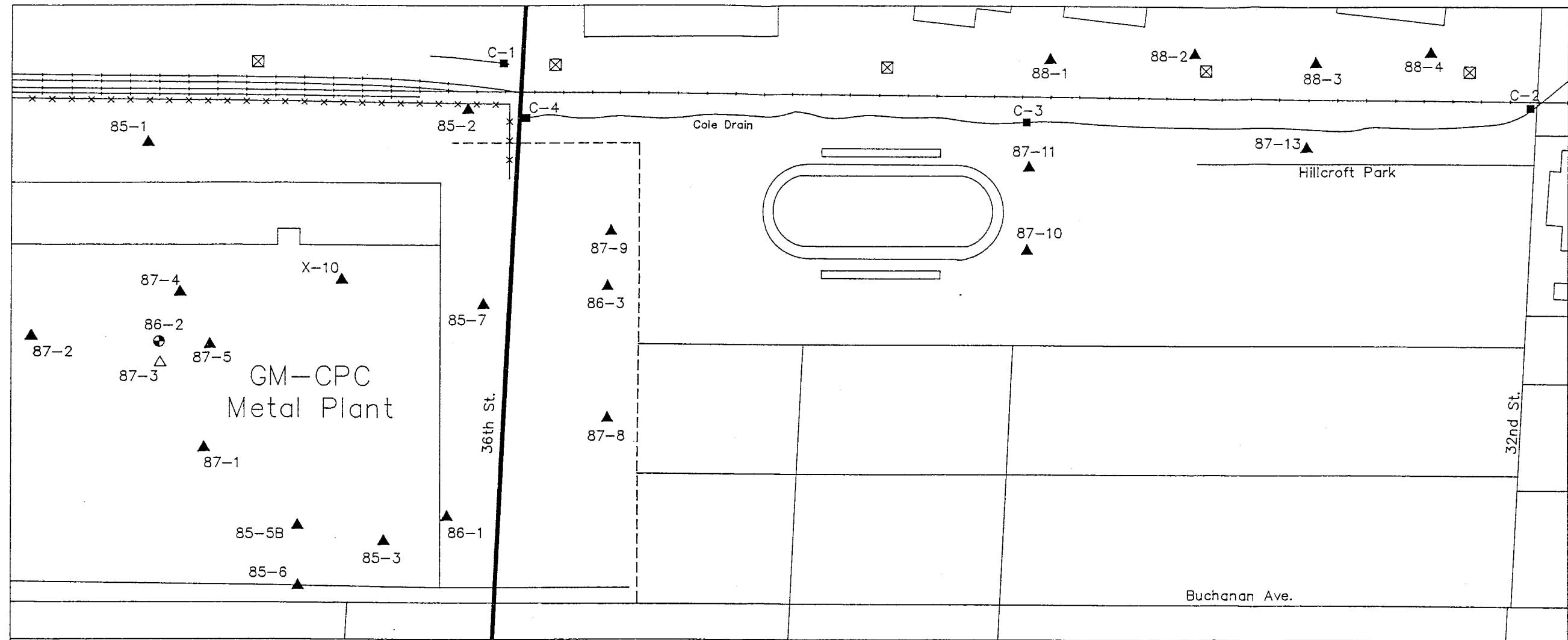
\* Matrix interference is not expected, but it could cause the detection limit to increase.


\*\* Includes cis- and trans-1,2-dichloroethylene

**TABLE 3**  
**MONITORING WELL AND STAFF GAUGE ELEVATIONS**

Well Site	Top of Casing (ft.)
85-1	675.61
85-2	672.22
85-3	681.11
85-5B	681.07
85-6	680.09
85-7	678.43
86-1	680.67
86-3	676.47
87-1	681.15
87-2	681.16
87-4	681.11
87-5	680.77
87-8	677.62
87-9	673.87
87-10	668.83
87-11	667.17
87-13	664.24
88-1	666.66
88-2	666.38
88-3	665.26
88-4	661.63
Vapor Well	
87-3	681.50
Purge Well	
86-2	681.12
Surface Site	Top Culvert (ft.)
C-1	663.20
C-2	657.02
C-4	664.45

**FIGURES**

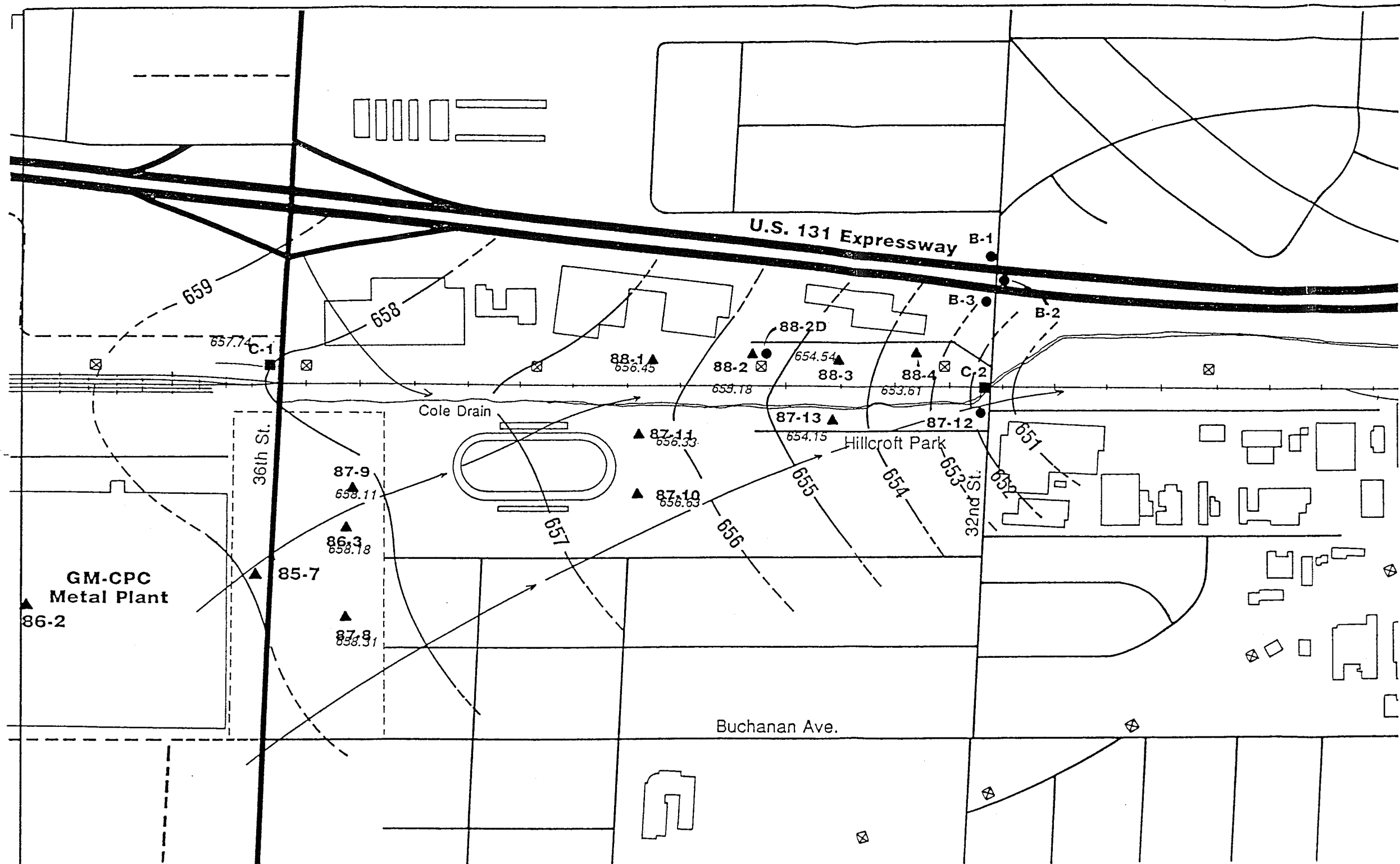


North 

0 100 200 300 600  
Scale in Feet

- ▲ Monitoring Well
- Purge Well
- △ Air Well
- ⊠ Electrical Tower
- Cole Drain Culvert
- x-x- Property Line

**FIGURE 1**  
**LOCATION OF WELLS  
 AND COLE DRAIN SITES**  
 GM CPC GROUP  
 GRAND RAPIDS, METAL FABRICATION PLANT  
 APRIL, 1988 21099



**LEGEND**

- ▲ Monitoring Wells
- Culverts Used to Measure Water Levels in Cole Drain
- Soil Borings Drilled During this Investigation (87-12, 88-2D)
- Soil Borings Drilled for US-131 Expressway During the 1950's (B-1, B-2, B-3)
- ⊠ Electrical Towers
- Direction of Groundwater Flow
- Contour Interval = 1ft.

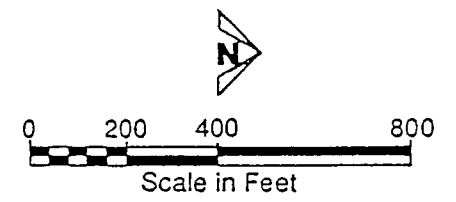
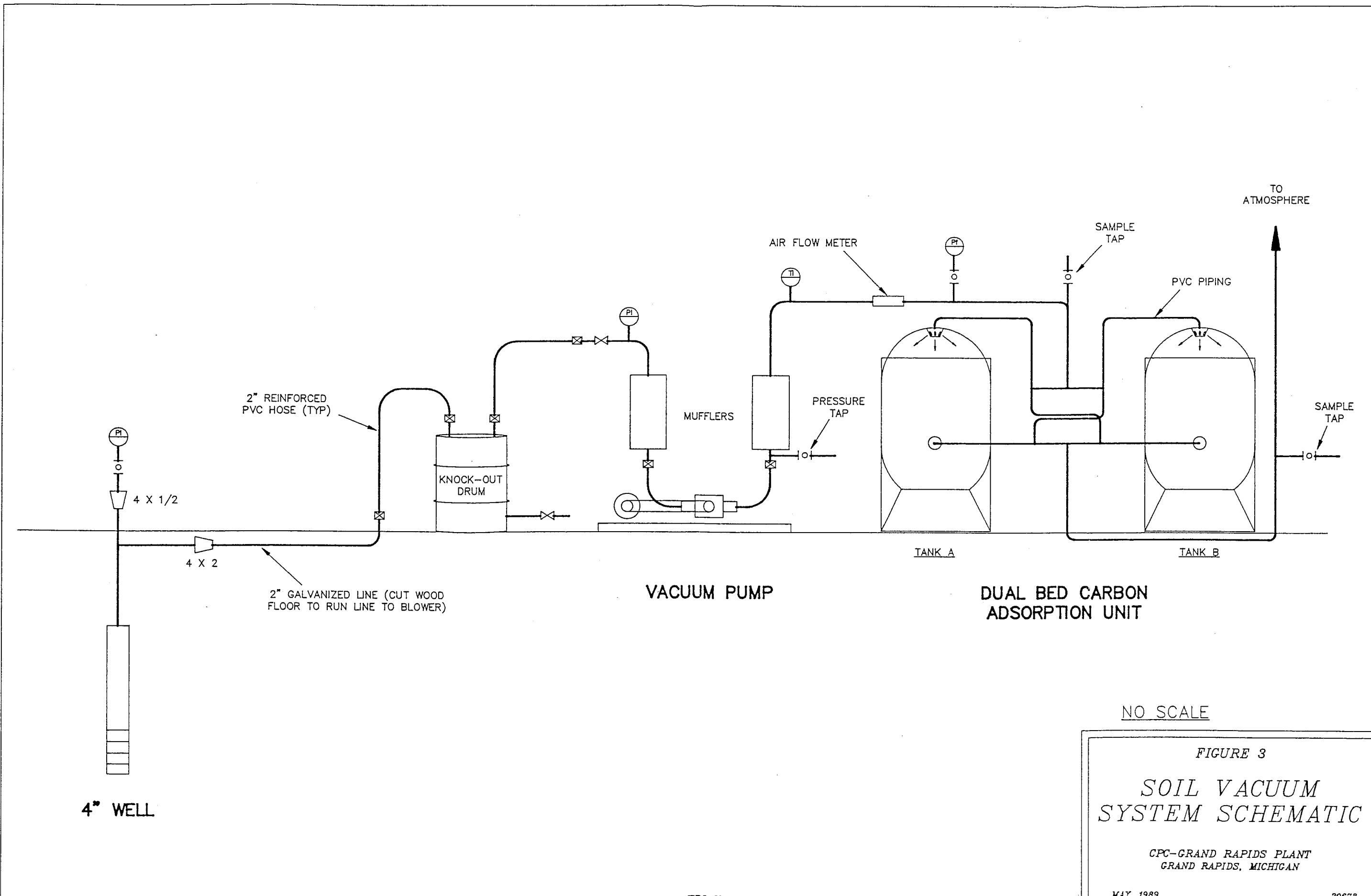
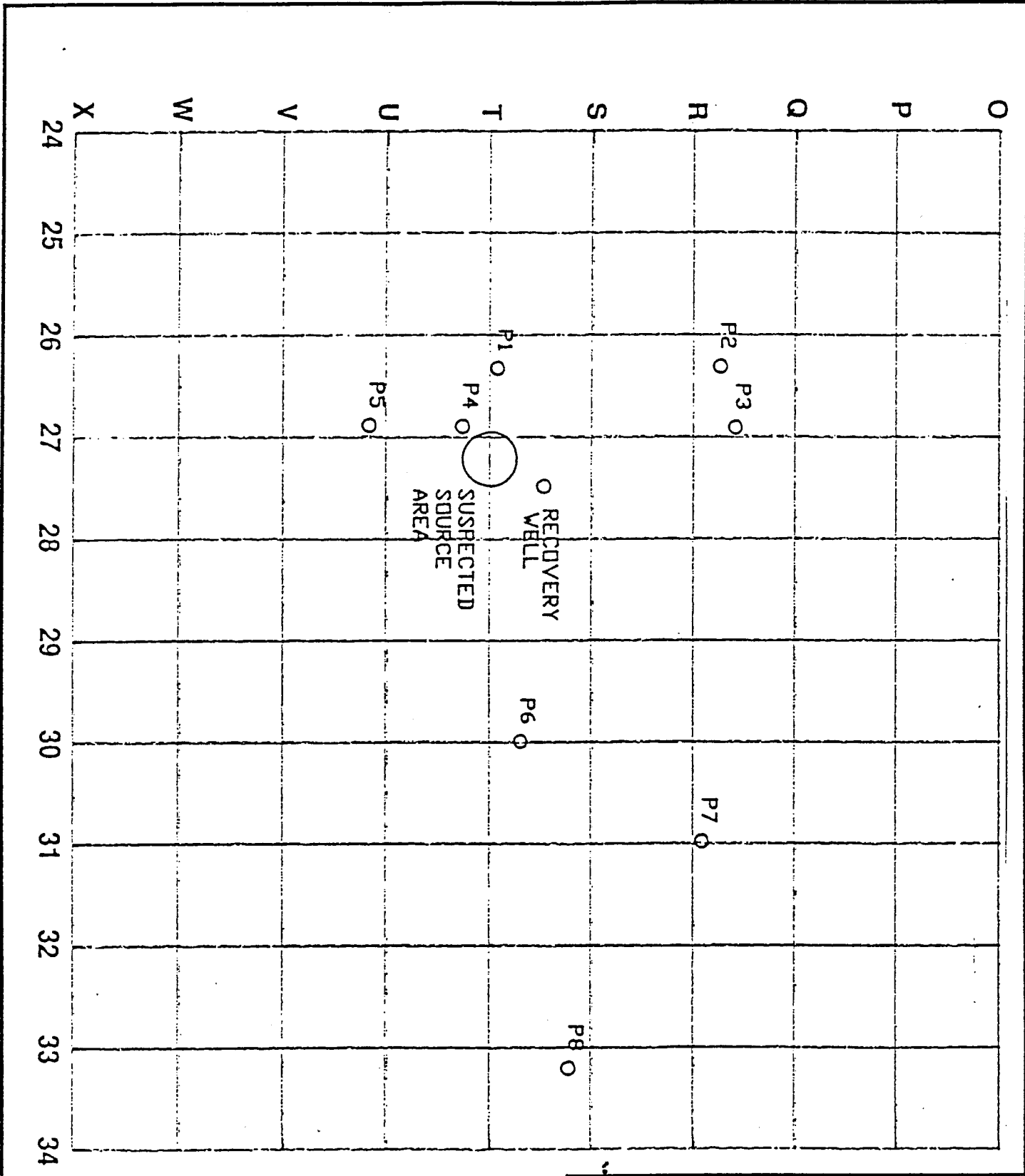


Figure 2  
**Water Table Contour Map**  
**April 7, 1988**  
 General Motors Corporation/CPC Group  
 Grand Rapids, Michigan



NO SCALE

FIGURE 3  
 SOIL VACUUM  
 SYSTEM SCHEMATIC  
 CPC-GRAND RAPIDS PLANT  
 GRAND RAPIDS, MICHIGAN  
 MAY, 1989 20673



SCALE 1" = 50'

Figure 4  
 Soil Vapor Probe Location  
 General Motors Corp./CPC Group  
 Grand Rapids, Michigan  
 August, 1988

**APPENDIX 1**

**WELL LOGS**









































**APPENDIX 2**  
**FIELD DATA ENTRY FORMS**

Date \_\_\_\_\_ Signature(s) \_\_\_\_\_

Monthly	WATER LEVEL	DEPTH TO WATER (ft.)	CHECK-OIL PRESENT (Y/N)	DEPTH OF WELL (ft.)	LENGTH OF WATER (ft.)	THREE CASING VOLUMES (gal.)	VOLUME PURGED (gal.)	DATE PURGED	TIME PURGED	DATE SAMPLED	TIME SAMPLED	THREE VIALS FILLED (Y/N)
85-1	X											
85-2	X											
85-3	X0											
85-5B	X0											
85-6	X0											
85-7	X											
86-1	X0											
PW86-2	X											
86-3	X											
87-1	X											
87-2	X											
87-4	X											
87-5	X											
87-8	X											
87-9	X											
87-10	X											
87-11	X											
87-13	X											
88-1	X											
88-2	X											
88-3	X											
88-4	X											
MW @ X-10	X											
PWdisch	X											
C-1/S36th	X											
C-2/32nd	X											
C-3/34th	X											
C-4/N36th	X											
FIELD BLANK												
TRIP BLANK												

Pressure readings (in.) P1 \_\_\_\_\_ P3 \_\_\_\_\_ P4 \_\_\_\_\_ P5 \_\_\_\_\_ 87-3 \_\_\_\_\_

Date \_\_\_\_\_ Signatures \_\_\_\_\_

Quarterly Sample	Water Level	Depth to Water (ft.)	Check-Oil Present (Y/N)	Depth of Well (ft.)	Length of Water (ft.)	Three Casing Volumes (gal.)	Volume Purged (gal.)	Date Purged	Time Purged	Date Sampled	Time Sampled	Three Vials Filled (Y/N)
85-1	X											
85-2	X											
85-3	X0											
85-5B	X0											
85-6	X0											
85-7	X											
86-1	X0											
PW86-2	X											
86-3	X											
87-1	X											
87-2	X											
87-4	X											
87-5	X											
87-8	X											
87-9	X											
87-10	X											
87-11	X											
87-13	X											
88-1	X											
88-2	X											
88-3	X											
88-4	X											
MW @ X10	X											
PWdisch	X											
C-1/S36th	X											
C-2/32nd	X											
C-3/34th	X											
C-4/N36th	X											
FIELD BLANK												
TRIP BLANK												

Pressure readings (in.) P1 \_\_\_\_\_ P4 \_\_\_\_\_ P5 \_\_\_\_\_ 87-3 \_\_\_\_\_

**APPENDIX 3**

**QUALITY ASSURANCE/QUALITY CONTROL  
FOR THE EDI ENGINEERING & SCIENCE LABORATORY**

EDI ENGINEERING & SCIENCE  
ANALYTICAL SERVICES DIVISION

QUALITY ASSURANCE/QUALITY CONTROL MANUAL

EDI ANALYTICAL SERVICES DIVISION  
QUALITY ASSURANCE/QUALITY CONTROL PROCEDURES MANUAL

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## 1.0 THE PURPOSE OF THE MANUAL

The purpose of this manual is to specify procedures and technical requirements to be used by the EDI Engineering & Science chemistry laboratories to assure that the data generated by the laboratory is accurate, reproducible and timely. This manual provides the chemistry laboratory a quality control plan which is to be used by every individual involved in the analytical efforts at EDI. This manual conforms to and is an extension of the EDI Quality Control Manual. If a conflict should arise, the EDI Corporate Quality Control Manual shall take precedence.

### 1.1 THE NEED FOR ANALYTICAL QUALITY CONTROL

There is a growing importance attached to the measurement of the concentration of any contaminant in water, effluents, and solid samples. As with any type of measurement the results of the methods utilized to measure the concentrations of these contaminants generally differ from the true concentration, i.e. all results are subject to error. Many experimental studies have shown that errors can arise which are as large as a 50 percent variations from the true value, and in fact, may vary between laboratories. Inaccurate analytical results restrict the ability of the analyst and the recipient of the data to draw valid conclusions and usually lead to false or misleading conclusions. Examples of common problems which arise during an analytical effort are as follows:

- A. Results, which are compared between two or more laboratories, are in error relative to each other.
- B. Results are to be used to decide if a water quality standard has been observed especially as the level of the analysis approaches the detection level.
- C. The analytical method, such as GCMS analysis, is only semi-quantitative in nature and, therefore, relative errors are anticipated between equivalent laboratories and even equivalent instruments.

- D. An inappropriate test procedure has been used to determine the analyte, resulting in values that do not represent the true sample concentration, i.e. direct aspiration of a turbid sample.

There is also increasing concern about the control of these errors being expressed at the local, the national and the international levels. The concern centers around the need to have a maximum amount of valid information obtained in a cost effective manner. In order to control errors, it is necessary to be able to measure the magnitude of these errors. This manual identifies the activities that are involved in the measurement and control of error. EDI considers analytical quality control of great importance, and requires that it be a primary feature in any analytical effort. The EDI requirements for analytical quality control are in concert with the quality control needs and demands of other organizations, such as the Environmental Protection Agency, the Michigan Department of Natural Resources, various state regulatory agencies, and such organizations as may need to insure their product quality.

Approximately twenty to thirty percent of all the available effort for routine analysis is absorbed in the execution of quality control requirements. It is often argued that the extent of this effort is too great with respect to routine laboratories and their need to be profitable in their operation. The argument therefore claims that extensive quality control is an impractical expectation for a routine laboratory. However, the corporate policy at EDI demands that the appropriate level of quality control be applied to all analytical effort at EDI, regardless of the sample lot.

In the total effort, it is preferable to obtain twenty to thirty percent fewer results of known accuracy for each analytical batch than it is to obtain larger numbers of results of undefined accuracy. Due to the fact that all analytical procedures are subject to errors derived from many sources, it is not reasonable to assume that quality control is unnecessary with a "good" analyst. However, even a "good" analyst may not have an adequate idea of his (her) accuracy. Multiple studies by the EPA, both within laboratories and between laboratories, has shown this reasoning to be generally unsound.

## 2.0 QUALITY ASSURANCE ORGANIZATION AND RESPONSIBILITIES

### 2.1 Objective of the QA Program at EDI

The purpose of the quality control program is to continuously monitor error, both random and systematic, which inhibits the production of reliable and defensive analytical data. Error is inherent in any analytical routine, even with the most rigorous controls, and thus, a good QC program addresses not only the basic control techniques but also statistical means of measuring precision and accuracy and the confidence limits on these measurements.

The purpose of this manual is to specify the procedures, records, Chain-of-Command, and technical requirements which will be adhered to by the laboratories of EDI. This manual conforms to and is an extension of the EDI Quality Assurance Manual and as such, gives final authority to the Corporate QA Manager.

### 2.2 Organization

Quality control at EDI begins with the bench analyst and moves up through the Chain of Command ultimately residing at the level of the President. A QC program which is administered only at the upper levels of management is doomed to failure and is unfair to the bench level analyst who needs a means by which he can observe the quality of his work. Quality Control is a two way program at EDI where directives from management are as important as suggestions and assistance from the bench analyst.

#### 2.2.1 QC Chain of Command Flow Chart

The following flow chart represents both the QA Chain of Command (solid line) and the Administrative Chain of Command (dotted). The flow chart represents the philosophy of EDI relative to the interaction of QC and production. Although the QC Coordinator reports to the Director of Analytical Services in the supervisory Chain-of-Command, his responsibilities for quality control require that

he answer to the Corporate QC officer. The QC coordinator acts as an immediate record keeper, QC administrator and liason to the lab manager.

However, when a question relative to the quality of analytical data arises, the QA coordinator, in conjunction with the Corporate QC Officer, has the right to prevent data dissemination. In cases of conflict, the Corporate QC Officer has final authority except when a compromise or directive is issued by the Chief Operating Officer or the President.

## 2.2.2 Responsibilities and Objectives of the Corporate QA Manager

### 2.2.2.1 Objectives

- o To develop and implement a Company wide quality assurance program
- o To assist management with the integration of individual quality assurance programs into all operations
- o To establish an attitude that focuses on defining causes of nonconformance and eliminating them
- o To assist managers in defining performance requirements

### 2.2.2.2 Responsibilities

The EDI Quality Assurance Manager (QAM) is responsible for developing and administering the EDI QA program. The QAM will conduct laboratory program audits and take appropriate action as necessary. This staff member will serve as the final authority in all laboratory QA programs. In addition, the QAM will serve as an arbitrator and final authority in all QA/QC problems which might arise in the

laboratory. A formal audit will be performed on the individual laboratories at least once per year by the QAM. The results of this audit will be submitted to the President. Prior to implementation, all QA/QC manuals and programs will be reviewed and approved by the QAM.

### 2.2.3 Responsibilities of the Laboratory QC Coordinator

- 2.2.3.1 To monitor the Quality Assurance activities in the laboratory insuring adherence to all policies and procedures.
- 2.2.3.2 To identify problem areas and help in recommending improvement and changes.
- 2.2.3.3 To keep abreast of changing development in analytical QC particularly requirements set by regulatory agencies.
- 2.2.3.4 To arrange or produce random blind control samples.
- 2.2.3.5 To approve all laboratory data prior to recording such data for report generation purposes.
- 2.2.3.6 To maintain QC on all analytical activities and update control limits in a timely manner.
- 2.2.3.7 Maintain balance and controlled temperature apparatus record books on a daily basis and insure that such records are maintained on every piece of equipment.
- 2.2.3.8 To assure that bottle preparation, approval and storage meet established criteria.

### 2.2.4 Responsibilities of the Sample Coordinator

A full position description of the Sample Coordinator can be found in the "EDI Log-in Manual".

- o To insure that all samples received at EDI are properly preserved, split, logged-in, and stored in agreement with the log-in manual.
- o To insure that all COC shipments are handled according to established procedures including storage, sample tracking and completion of files.
- o To insure that all project sheets and subsequent paperwork is completed and filed.
- o To insure that labile samples are distributed in a timely manner.
- o To cooperate with the QC coordinator in introducing blind samples.

#### 2.2.5 Responsibilities of the Analytical Staff

- o To insure that all records are generated and recorded on a daily basis.
- o To insure that the following bench level QC requirements are met.
- o To fill out lab notebooks daily is required.
- o To provide for QC on every batch of samples at a minimum of
  - 10% duplicate
  - 10% blanks
  - 10% matrix spike
  - method spikes, at least one per set of samples
- o To insure that instruments are calibrated prior to initiating any analysis and that no analyses are started unless calibration has been satisfactorily completed.

- o To insure that every batch of analyses meets established QC guidelines or is reanalyzed automatically.

- o To inform the lab manager of any reoccurring problems or systematic trends which may effect quality.

#### 2.2.6 Responsibilities of the Laboratory Supervisor

- o To insure that sufficient competent staff is available to administer QC.

- o To insure that all participating analysts are certified in the test they are performing.

- o To insure that effective training and orientation takes place for every new analyst.

- o To insure that all QC procedures, directives or project oriented requirements are met.

- o To review all preliminary reports and approve them prior to the generation of a final report.

- o To interface with the QC coordinator and corporate QC Manager on a routine and consistent basis.

- o To take responsibility for immediate solutions to QC problems which may slow or stop production.

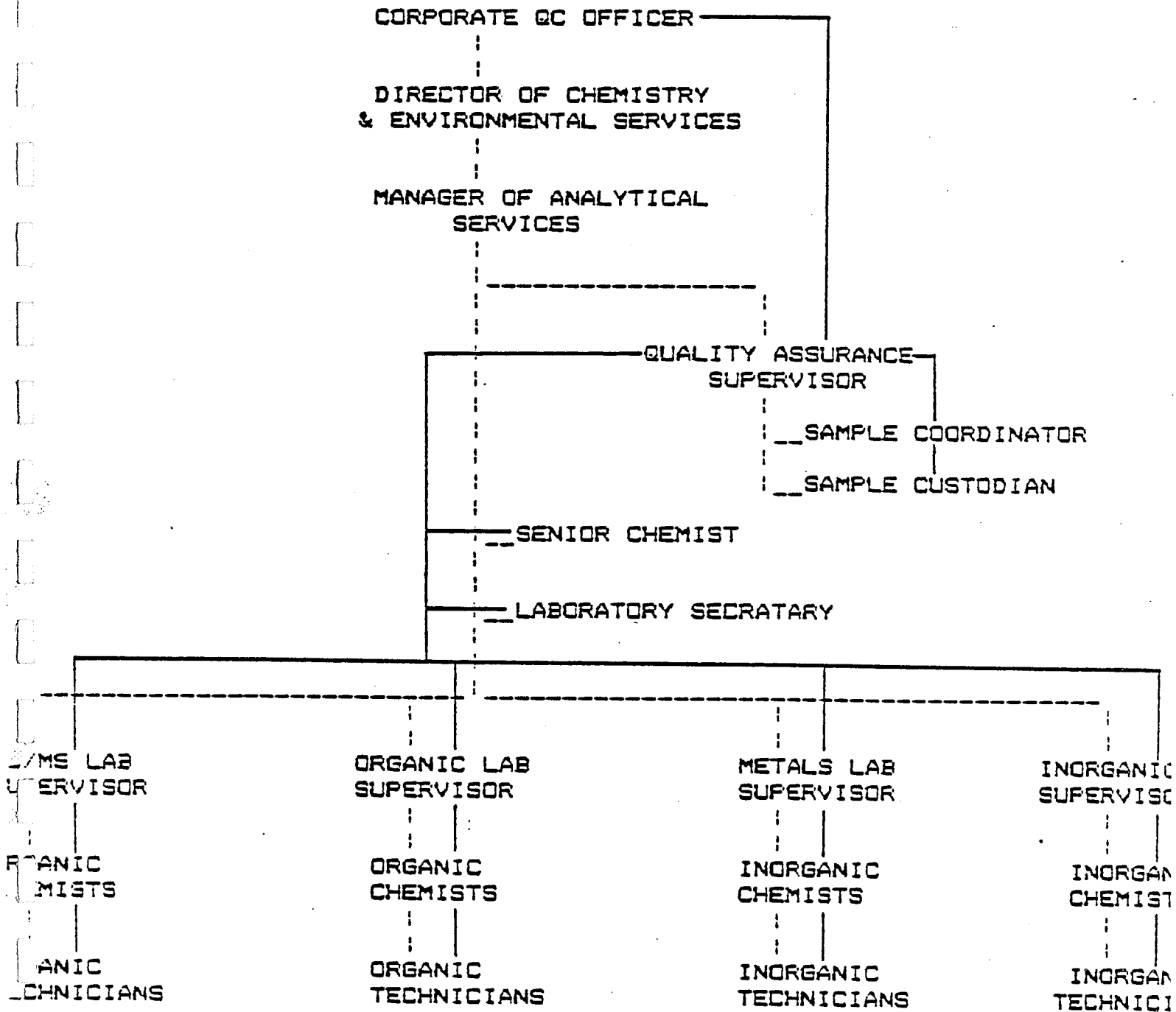
#### 2.2.7 Responsibilities of the Data Coordinator

The Data Coordinator's (DC) responsibilities are:

- o To enter all data generated into the appropriate records.

- o To insure that the QC Coordinator has signed the data forms (bench sheets prior to entry).
- o To inform the QC Coordinator when a project is complete and ready for a preliminary report.
- o To provide corrections to all reports from preliminary report feedback.

# QUALITY CONTROL CHAIN OF COMMAND FLOW CHART



ADMINISTRATIVE FLOW = \_\_\_\_\_  
 QUALITY CONTROL FLOW = \_\_\_\_\_

### 3.0 FACILITIES AND EQUIPMENT

- 3.1 The physical plant layout diagram is enclosed. The approximate square footage allocated to each analysis area is presented as well as the number of personnel normally working in each area. A listing of equipment presently utilized by EDI is also enclosed.
- 3.2 The quality of the analytical instrumentation utilized by EDI is of great importance considering its ultimate effect on data quality. The following guidelines exist for the procurement of analytical instrumentation:

#### 3.2.1 Equipment Need

An equipment need is identified by the Lab Manager or the Director of Environmental Services as a result of:

- o New Contractual Effort
- o Regulatory Changes
- o Normal Upgrade/Replacement
- o Capacity Improvements

#### 3.2.2 Procurement Procedure

The performance specifications defined by the need are used to identify prospective equipment suppliers. The Lab Manager mails the performance specifications to the prospective equipment suppliers. Those suppliers able to meet the performance specifications are asked to provide a quotation for the purchase or lease of the equipment. An evaluation of the quotations is made by the Lab Manager with consideration given to such items as: equipment ease of use, degree of automation, specification compliance, potential for computerization, price and space requirements.

A written recommendation by the Lab Manager is presented to the Director of Environmental Services and the Quality Assurance Office for their review and comment.

A final recommendation by the Director of Environmental Services is made to the President of EDI and the Corporate Quality Assurance Officer. The final approval is granted based on the assurance of complying with all regulatory and corporate guidelines for the generation of the highest quality data.

### 3.3 Chemical Procurement and Inventory Procedure

All reagent specifications are dictated by the EPA/APHA or NIOSH approved analytical methods. These reagent specifications are identified and maintained on the chemical inventory index card system. The chemical inventory index card system assures the order of chemical use and minimizes the possibility of exceeding their useful shelf life. The addition of a new method or a change in an existing method that requires a corresponding addition or change in a reagent used for that method will be identified by the Lab Area Supervisor or Group Leader. The Supervisor or Group Leader will prepare the chemical inventory index.

All reagent specifications, available vendors and amount(s) received are recorded on the chemical inventory index cards.

All chemical reagents are received by the sample coordinator. The sample coordinator notifies a designated laboratory aid that a delivery has arrived. The aid opens the shipping package and compares the packing slip with the contents. Discrepancies are identified to the Lab Manager. The materials receipt is identified and recorded on the chemical inventory. The materials are then inventoried on the chemical inventory index. The index system identifies the amount(s) received and when. When the last bottle/container of the chemical remains, the chemical is placed on an open order sheet located in the lab.

The group leader/supervisor for that lab area is responsible for picking up the chemical open order sheet each week and preparing a purchase order. The purchase order is approved by the Lab Manager and a typed purchase order is issued to the approved vendor that has been previously identified as being able to supply the specified material. The receipt of the new order initiates the inventory system activities.

#### 3.4 Preventative Maintenance

Every analytical instrument has a separate maintenance log book as identified in the Document Control Section No. 7.3.9. The required maintenance activities have been developed by the Lab Manager and each Group Leader/Area Supervisor. The maintenance activities comply with manufacturer specifications and working experience requirements.

Each maintenance log book contains a table indicating the frequency and type of maintenance required. The maintenance activity is documented each day or as the frequency requirements dictate.

Analysts are assigned the responsibility of maintaining various instruments or equipment in their respective laboratory areas. The Group Leader or Area Supervisor is responsible for checking the maintenance log books each week and signing off as checked. The Quality Assurance Officer is notified of any deviations or lack of maintenance activity performance, and corrective actions are taken.

EQUIPMENT

ANALYTICAL AREA	EQUIPMENT/ INSTRUMENT NAME	MANUFACTURER	MODEL NO.
Wet Chemistry	Analytical Balance	Mettler	AE163
	Auto-analyzer	Alpkem (refurbished Technicon	
	Beckman pH meter	Beckman-Altex	070
	Spectrophotometer	Hitachi	100-40
	Conductivity Meter	HACH	portable
	Dri-Block-digestor	Tecam	DB-3H
Atomic Absorption	Atomic absorption Spectrophotometer	Varian Perkin-Elmer	AA-575 5000
	Gas Chromatography	Analytical balance	Mettler
Gas Chromatograph #1		Varian	3700
Gas Chromatograph #2		Varian	3760
Gas Chromatograph #3		Varian	3760
Gas Chromatograph #4		Varian	3400
Integrators (3)		Varian	4270
Chart recorder (2)		Various	
GC/MS Spectrometry	Gas chromatography/ Mass spectrometer	Finnegan MAT	OWA 20B
	Gas chromatography/ Mass spectrometer	Finnegan MAT	OWA 20B
	Nine Track Magnetic Tape System	Data General	6021

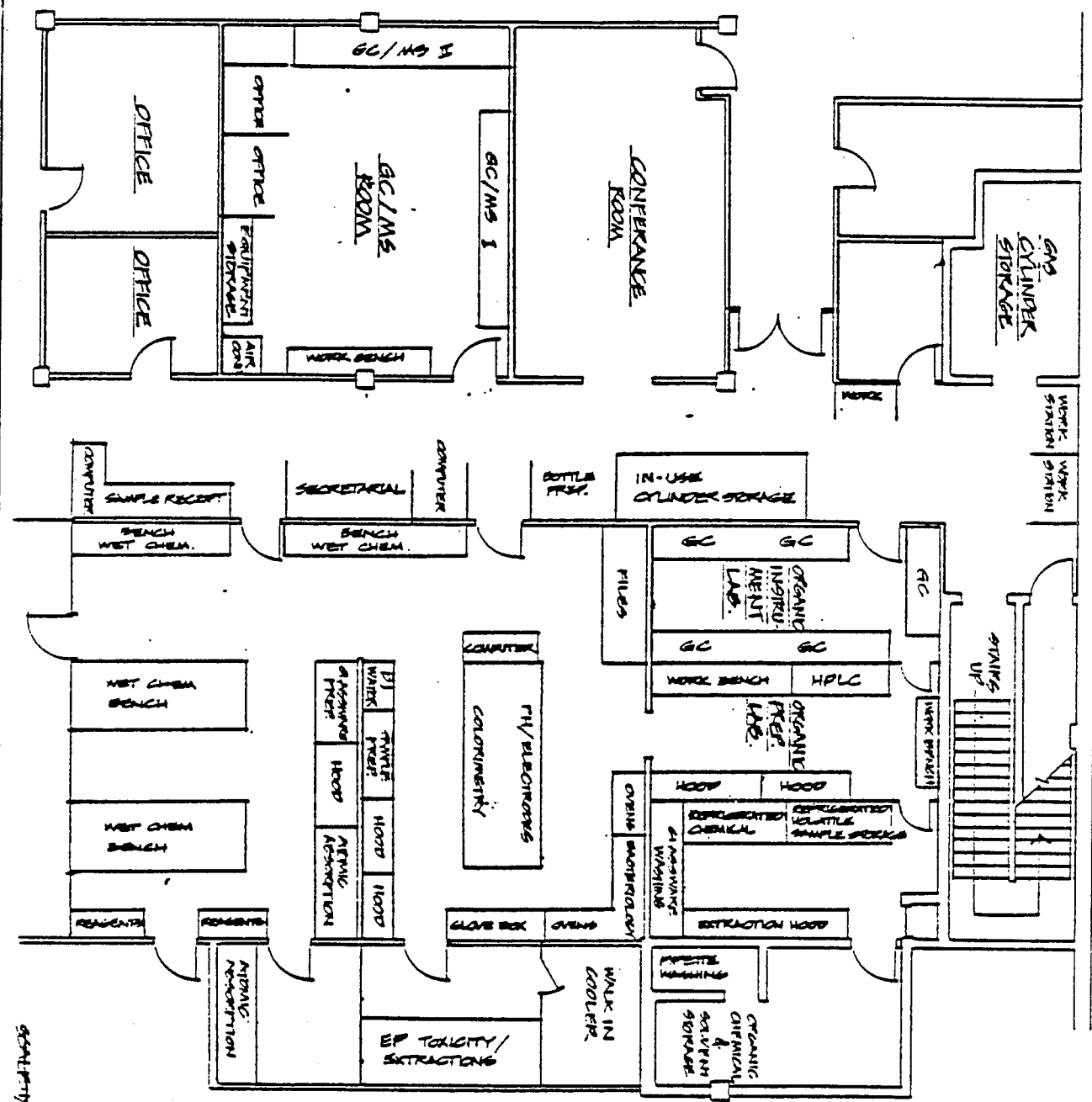
PHYSICAL PLANT

Laboratory Name and Address: EDI ENGINEERING & SCIENCE  
611 Cascade West Parkway S.E.  
Grand Rapids, MI 49506

Name of Laboratory Manager: John P. Dullaghan

Describe or attach a drawing of the laboratory layout, indicating the general areas of analysis, the space allotted to each and the number of personnel generally assigned to each area.

ANALYSIS	SPACE ALLOTTED, FT <sup>2</sup>	NUMBER OF PERSONNEL
MICROBIOLOGY	Approx. 30	1
WET CHEMISTRY	" 970	5
ATOMIC ABSORPTION/EMISSION	" 300	1
GAS CHROMATOGRAPHY (GC)	" 380	3
GC/MASS SPECTROMETRY	" 500	3
SAMPLE RECEPTION	" 100	1
ADMINISTRATIVE (TYPING, ETC.)	" 400	4
STORAGE (CYLINDERS, WASTES)	" 400	1



SCALE: 1/8" = 1'-0"

EDI ENGINEERING & SCIENCE  
 1000 WEST 10TH AVENUE, SUITE 1000  
 DENVER, CO 80202  
 ANALYTICAL LABORATORY

## 4.0 ANALYTICAL METHODOLOGIES

### 4.1 Methods Utilized

The EDI Laboratory maintains and updated reference volumes of approved analytical methodologies for environmental and non-environmental analysis. A responsibility of the Lab Manager and the Director of Environmental Services is to continually seek and review regulatory method changes and their impact on current laboratory practices. The most commonly referenced materials include:

- o "Methods for Chemical Analysis of Water and Wastes" EPA-600/4-79-020 revised March 1972.
- o "Manual of Analytical Procedures" NIOSH, Volumes 1 & 2, Third Edition Feb., 1984.
- o Standard Methods for the Evaluation of Water and Wastewater, APHA, AWWA, WPCF 1980.
- o "Handbook for Analytical Quality Control in Water and Wastewater Laboratories", EPA 600/4-79-019, March 1979.
- o "Physical and Chemical Methods for the Evaluation of Solid Waste" EPA-SW846 Second Edition, 1982.
- o "Guidelines Establishing Text Procedures for the Analysis of Pollutants" Proposed Regulations. CFR December 3, 1979; updated Oct. 26, 1984.

### 4.2 Method Calibration and Operating Procedures

All methods utilized by EDI have been certified in accordance with the specifications outlined in the Method Certification Section No. 5.0.

A standard operating procedure manual exists for all analytical procedures. The S.O.P.'s include specific calibration procedures that must be followed by an analyst prior to conducting sample analysis. The analyst is

required to perform and document the calibration procedure. The calibration activity is identified by each analyst in their lab notebooks. The actual standards utilized and resulting measurements are found in each instrument log book. It is the responsibility of each analyst to document all calibration and operating procedures utilized, in their personal log books as well as in the instrument log books. The area group leaders and/or supervisors review this information each week when they sign off indicating the information has been reviewed. It is the responsibility of the group leaders and/or supervisors to notify the Quality Assurance Officer when deviations occur so that corrective actions can be taken. The corrective action will be to identify whether the information is simply missing (not entered) and to have it recorded or if the calibration has not been performed, to not release data generated that day and require those samples to be rerun.

## 5.0 METHOD CERTIFICATION

All methods used by EDI which were not developed by EDI will be certified prior to their use. Method Certification is contiguous with the certification of the analyst and requires essentially the same analytical program. Method certification is necessary in order to establish detection limits, method application limits and criteria for control limits. In most cases, detection limits and recoveries stated in a method are obtained under ideal conditions and do not reflect real world solutions, e.e. silty well water and industrial effluent versus a drinking water supply. Method certification falls into 2 categories: 1) Methods being employed for the first time and 2) Methods which are to replace currently certified methods (replacement methods). In either case, analysis of client sample may not proceed until certification has occurred.

### 5.1 Method Certification

#### 5.1.1 Linear Range

The first step in certifying a method is to establish the linear range (operating range) of the method. A method may be used only over the range in which it is linear. Some methods do not have linear ranges but curves from which results are calculated. For the moment we will ignore methods with curves. A linear range must be established independent of the method data since instruments can effect the range. Standards and multiple detections will be used for establishing the linear range. For example, a range of 1 to 1000 has 3 decades (3 orders of magnitude or  $10^3$ ). Therefore, a range of 1 to 1000 requires 11 levels of test standards (.5, 1, 2, 5, 10, 20, 50, 100, 200, 500, 1000). Notice that each decade follows the 0.5x to 10x rule, i.e. the area 10 to 100 is covered by 5, 10, 20, 50 and 100. The range to be attempted is dependent on the method, the instrument and the analytical supervisor. If the responses show linearity, the range has been established. If,

however, a curve develops or there appear to be two linear ranges, the standards must be repeated including additional levels to verify the status of the questionable area.

#### 5.1.2 Working Curves

Some methods operate from a curve response, i.e. sodium by emission spectroscopy. The method will indicate the working curve which must be verified. The method with working curves requires a full curve each time an analysis is to be performed.

#### 5.1.3 The Generation of the Method Detection Limit

The method detection limit (MDL) is defined as the minimum concentration of a substance that can be identified, measured and reported with 99% confidence that the analyte concentration is greater than zero and determined from analysis of a sample in a given matrix containing analyte.

##### Scope and Application

This procedure is designed for applicability to a wide variety of sample types ranging from reagent (blank) water containing analyte to wastewater containing analyte. The MDL for an analytical procedure may vary as a function of sample type. The procedure requires a complete, specific and well defined analytical method. It is essential that all sample processing steps of the analytical method be included in the determination of the method detection limit.

The MDL obtained by this procedure is used to judge the significance of single measurement of a future sample.

The MDL procedure was designed for applicability to a broad variety of physical and chemical methods. To accomplish this, the procedure was made device or instrument independent.

## Procedure

1. Make an estimate of the detection limit using one of the following:
  - (a) The concentration value that corresponds to an instrument signal/noise ratio in the range of 2.5 to 5. If the criteria for qualitative identification of the analyte is based upon pattern recognition techniques, the least abundant signal necessary to achieve identification must be considered in making the estimate (PCB).
  - (b) The concentration value that corresponds to three times the standard deviation of replicate instrumental measurements for the analyte in reagent water.
  - (c) The concentration value that corresponds to the region of the standard curve where there is a significant change in sensitivity at low analyte concentrations i.e. a break in the slope of the standard curve.
  - (d) The concentration value that corresponds to known instrumental limitations.

It is recognized that the experience of the analyst is important to this process. However, the analyst must include the above considerations in the estimate of the detection limit.

2. Prepare reagent (blank) water that is as free of analyte as possible. Reagent or interference free water is defined as a water sample in which analyte and interferent concentrations are not detected at the method detection limit of each analyte of interest. Interferences are defined as systematic errors in the measured analytical signal of an established procedure caused by the presence of interfering species (interferent). The interferent concentration is presupposed to be normally distributed in representative samples of a given matrix.

3. (a) If the MDL is to be determined in reagent water (blank) prepare a laboratory standard (analyte in reagent water) at a concentration which is at least equal to or in the same concentration range as the estimated MDL (Recommend between 1 and 5 times the estimated MDL) Proceed to Step 4.
- (b) If the MDL is to be determined in another sample matrix, analyze the sample. If the measured level of the analyte is in the recommended range of one to five times the estimated MDL proceed to Step 4.
- If the measured level of analyte is greater than five times the estimated MDL, add a known amount of analyte to bring the concentration of analyte to between one and five times the MDL in the case where an interference is coanalyzed with the analyte.
- If the measured level of analyte is greater than five times the estimated MDL there are two options:
- (1) Obtain another sample of lower level of analyte in same matrix if possible.
  - (2) The sample may be used as is for determining the MDL if the analyte level does not exceed 20 times the MDL of the analyte in reagent water. The variance of the analytical method changes as in the analyte concentration increases from the MDL, hence the MDL determined under these circumstances may not truly reflect method variance at lower analyte concentrations.
4. (a) Take a minimum of seven aliquots of the sample to be used to calculate the MDL and process each through the entire analytical method. Make all computations according to the defined method with final results in the method reporting units. If blank measurements are required to calculate the measured level of analyte, obtain separate blank measurements for each sample aliquot analyzed. The average blank measurement is subtracted from the respective sample measurements.

(b) It may be economically and technically desirable to evaluate the estimated MDL before proceeding with 4a. This will: (1) prevent repeating this entire procedure when the costs of analyses are high and (2) insure that the procedure is being conducted at the correct concentration. It is quite possible that an incorrect MDL can be calculated from data obtained at many times the real MDL even though the background concentration of analyte is less than five times the calculated MDL. To insure that the estimate of the MDL is a good estimate, it is necessary to determine that a lower concentration of analyte will not result in a significantly lower MDL. Take two aliquots of the sample to be used to calculate the MDL and process each through the entire method, including blank measurements as described above in 4a. Evaluate these data:

- (1) If these measurements indicate the sample is in the desirable range for determining the MDL, take five additional aliquots and proceed. Use all seven measurements to calculate the MDL.
- (2) If these measurements indicate the sample is not in the correct range, reestimate the MDL, obtain new sample as in 3 and repeat either 4a or 4b.

5. Calculate the variance ( $S^2$ ) and standard deviation ( $S$ ) of the replicate measurements, as follows:

$$S^2 = \frac{1}{n-1} \left[ \sum_{i=1}^n X_i^2 - \frac{(\sum_{i=1}^n X_i)^2}{n} \right]$$

$$S = \sqrt{S^2}$$

where the  $x_i$   $i = 1$  to  $n$  are the analytical results in the final method reporting units obtained from the  $n$  sample aliquots and

$X_i^2$  refers to the sum of the X values from  $i = 1$  to  $n$ .

6. (a) Compute the MDL as follows:

$$MDL = t_{(n-1), 1-\alpha=.99} \cdot S$$

where:

MDL - the method detection

$t_{(n-1), 1-\alpha=.99}$  = the students' t value appropriate for a 99% confidence level and a standard deviation estimate with  $n-1$  degrees of freedom. See Table.

S = standard deviation of the replicate analyses.

- (b) The 95% confidence limits for the MDL derived in 6a are computed according to the following equations derived from percentiles of the chi square over degrees of freedom distribution ( $\chi^2/df$ ) and calculated as follows:

$$MDL_{lcl} = 0.69 MDL$$

$$MDL_{uc1} = 1.92 MDL$$

where  $MDL_{lcl}$  and  $MDL_{uc1}$  are the lower and upper 95% confidence limits respectively based on seven aliquots.

7. Optional iterative procedure to verify the reasonableness of the estimated MDL and calculated MDL of subsequent MDL determinations.

- (a) If this is the initial attempt to compute MDL based on the estimated MDL in Step 1, take the MDL as calculated in Step 6, spike in the matrix at the calculated MDL and proceed through the procedure starting with Step 4.

- (b) If the current MDL determination is an iteration of the MDL procedure for which the spiking level does not permit qualitative identification, report the MDL as that concentration between the current spike level and the previous spike level which allows qualitative identification.
- (c) If the current MDL determination is an iteration of the MDL procedure and the spiking level allows qualitative identification, use  $S^2$  from the current MDL calculation and  $S^2$  from the previous MDL calculation to compute the F ratio.

$$\text{if } \frac{S_A^2}{S_B^2} < 3.05$$

then compute the pooled standard deviation by the following equation:

$$\text{Spooled} = \left[ \frac{6S_A^2 + 6S_B^2}{12} \right]^{1/2}$$

if  $\frac{S_A^2}{S_B^2} > 3.05$ , respike at the last calculated MDL and process the samples through the procedure starting with step 4.

- (c) Use the Spooled as calculated in 7b to compute the final MDL according to the following equation:

$$\text{MDL} = 2.681 (\text{Spooled})$$

where 2.681 is equal to  $t_{(12, 1-\alpha = .99)}$

- (d) The 95% confidence limits for MDL derived in 7c are computed according to the following equations derived from percentiles of the chi squared over degrees of freedom distribution.

$$\text{MDL}_{\text{lcl}} = 0.72 \text{ MDL}$$

$$\text{MDL}_{\text{uc1}} = 1.65 \text{ MDL}$$

where LCL and UCL are the lower and upper 95% confidence limits respectively based on 14 aliquots.

### Reporting

The analytical method used must be specifically identified by number or title and the MDL for each analyte expressed in the appropriate method reporting units, if the analytical method permits options which affect the method detection limit these conditions must be specified with the MDL value. The sample matrix used to determine the MDL must also be identified with the MDL value. Report the mean analyte level with the MDL if a laboratory standard or a sample that contained a known amount analyte was used for this determination, report the mean recovery, and indicate if the MDL determination was iterated.

If the level of the analyte in the sample matrix exceeds 10 times the MDL of the analyte in reagent water, do not report a value for the MDL

### Reference

Glaser, J.A. Foerst, D.L. McKee, G.D. Quave, S.A. and Budde, W.L. "Trace Analysis for Wastewater, "Environmental Science and Technology, 15, 1426 (1981)

Table of Students  $t$  Values at the 99 Percent Confidence Level

Number of Replicates	Degrees of Freedom (n-1)	$t_{(n-1), 1-\alpha=99}$
7	6	3.143
8	7	2.998

Table of Students'  $t$  Values at the 99 Percent Confidence Level (Cont'd)

Number of Replicates	Degrees of Freedom ( $n-1$ )	$t_{(n-1), 1-\alpha=.99}$
9	8	2.896
10	9	2.821
11	10	2.764
16	15	2.602
21	20	2.528
26	25	2.485
31	30	2.457
61	60	2.390
		2.326

#### 5.1.4 Method Spikes

Method spikes will be carried out over 2 separate days at the specified levels including blanks and calibration standards. The data obtained at the 2x or 5x level (for each certified range) will be used to establish a mean and standard deviation for initial control charts. Once this data has been generated and approved by the analytical manager, the method has preliminary certification and is ready for application to real world samples. These control limits will be updated with every batch of samples until 30 numbers have been developed to establish reliable control limits. After 30 data points the DC will provide updated control limits with each additional 20 numbers.

#### 5.2 Replacement Method Certification

When a new method is to be employed (where a new method is defined as including a new instrument method, i.e. flame vs flameless AA or Hall vs ECD), the method must be certified prior to its use on client samples.

Certification follows the procedures described in Sections 5.1. The results of these tests are important but are not necessarily compared to the current method. The detection limit may change and the work range may change but if they meet the needs of the lab, these changes are to be ignored. One may elect to utilize a t-test analysis to identify the method differences as being significant or not.

#### 5.2.1 Comparison by the t-Test

One sample will be analyzed a minimum of 4 times by each method. The results will be subject to a t-test analyses. If the t-test indicates statistical correlation, regardless of the correlation coefficient, the new method is certified. If the t-test fails, refer to Section 5.2.2 below.

#### 5.2.2 Decisions on Certification

The purpose of a new method is to improve accuracy, precision and efficiency. Efficiency is of no consequence if a method is imprecise and inaccurate, and therefore, is not a consideration in certifying new methods. However, a new method may fail the t-test because it is more accurate and/or more precise. Careful consideration and more analyses may be necessary with a new method by the analytical manager and supervisor.

## 6.0 ANALYST TRAINING AND CERTIFICATION

### 6.1 Rational

Consistent with requirements by the EPA and other regulatory agencies for analyst training and certification programs, EDI has a strict policy relative to the training and certification of analysts prior to their involvement in the analysis of client samples. The program is necessary in order to maintain continuity in all analytical programs and to insure the integrity of all data.

### 6.2 Training

The supervisor is responsible for training all new personnel. This training will be in conjunction with the group (workstation) and group leader if applicable. Training will include, but not be limited to, EDI QC requirements, paperwork flow, lab safety and organizational structure. In addition, the new analyst will be given copies of the QC manual, log-in manual and methodologies which the analyst will be required to read. Training in the methods to be used will be initiated prior to analyst certification.

### 6.3 Certification

Each new EDI analyst will be required to receive certification on all methods which he is to perform. Certification insures that the analyst can meet EDI detection limits and quality control limits as established for the method. Certification includes two parts, both of which must be completed satisfactorily.

#### 6.3.1 Method Spikes

Analysis of spiked lab pure water at the levels of 0.5x, 1.0x, 2.0x, 5.0x and 10x where x is the established detection limit. This will include 2 blanks and a duplicated spike at 2.0x or 5.0x and will occur on 2 separate days. The data, where the duplicated

results are averaged. These results must match current EDI Schwart control chart limits. Additional parameters such as consistent instrument calibration curves will be evaluated.

#### 6.3.2 Check Sample Analysis

The analyst will test a known blind check sample in duplicate including a blank. All the data must fall within established control limits for the parameters.

#### 6.3.3 Current Analysts Training

The EDI analyst, who is assigned a new method, must complete the certification program for the methods as outlined above prior to performing analyses on client samples.

#### 6.4 Recertification

All EDI analysts will recertify on all their respective methods when required or demonstrated by two method spike performance failures following the procedures set forth in Section 6.3.1. The results must meet previous data, assuming that the same methods are employed.

#### 6.5 Performance Audits

The Analytical Manager, in cooperation with the Data Coordinator and Corporate QC Manager, will perform individual audits on all aspects of the operation biannually. These audits will include recertification data, control limits, all levels of records and laboratory performance on all check samples and instituted blind QC samples. A report of the audit results including recommendations will be forwarded to the President of EDI.

## 7.0 DOCUMENT CONTROL, FLOW AND STORAGE

### 7.1 Purpose

The paperwork trail must be designed to insure that after the issuance of a report, anyone - a client, a lawyer or the President of EDI - can track a single sample result back through EDI records to the origin of the standards used in calibration and the identity of the person who prepared the sample bottles.

### 7.2 Paperwork Flow

As shown in Figure 1, "Flow Diagram" the paperwork trail is eventually the same for routine work as it is for samples under Chain-of-Custody. The general axiom is that a COC procedure is doomed to failure without a pre-existing scheme of tight sample and analytical control available as a routine measure. This contention, however, is only of minimal consequence with respect to the need for detailed records. The records trail can provide the following:

- o Answers to questions of analytical integrity for results which are 2 months or two years old.
- o Assistance in finding and solving random and systematic problems.
- o Assistance in preventing long term degradation of analytical integrity.
- o Assistance in insuring continuity of analytical effort despite personnel and mechanical changes.

### 7.3 Document Requirements

The following subsection identifies all documents which are generated during the course of any project:

### 7.3.1 Project Sheets

Every sample or group of samples which enter the EDI facility must be accompanied by the appropriate project sheet which has been properly filled out and provided to the Sample Coordinator (SC). The SC may not log-in samples for which there are not project sheets or for which there the project sheet is incomplete. An example project sheet is attached as Figure 2.

### 7.3.2 New Project Approval Form

Projects which require testing or analyses not routinely provided at EDI must have prior approval on a "EDI Project Approval Form" and commitment from the Analytical Manager and the head of the appropriate analytical group(s). For the project manager's purpose, the approval forms insure that the analytical testing area has received notification and will be prepared. For the analytical managers purpose, proper notification has been received and sufficient time has been allotted for preparation and development. Projects requiring rush turn around on modified methods must be approved as well. An example of a New Project Approval Form is attached as Figure 3.

### 7.3.3 Problem Project Sheets

When the Sample Coordinator (SC) identifies a problem with a sample shipment or project sheet, a Problem Project Sheet will be initialed and sent to the project manager for resolution. See Figure 4.

### 7.3.4 Chain-of-Custody Forms

There are three forms for Chain-of-Custody samples. All three

forms must be properly completed and included in the project file for each and every COC project.

#### 7.3.4.1 COC Shipping Record

The shipping record must be received in the shipping container with every COC shipment. The form attached as Figure 5 is identical to the form used by the EPA. This form will be used by EDI field samplers and returned with the samples. Other forms of a similar nature may be used by other clients. However, the information required on the EDI form must be present on any other client form or they run the risk of their COC being rejected as a continuous trackable COC event.

#### 7.3.4.2 COC Project Log-in Form

This form, attached as Figure 6, is to be used for the receipt of any COC shipment regardless of the origin of the shipping record. COC projects which have poor or nonexistent shipping records will be logged-in (with notification of the deficiency to the Project Manager) and this form will be completed. The form is completed by the Sample Coordinator and placed immediately in the COC Project File.

#### 7.3.4.3 COC Sample Control Record

This form is used as a record of the movement of COC samples in and out of the COC locked storage. The analyst signs samples in and out each time a sample(s) is removed for any analysis. A copy of the form is attached as Figure 7. After all analyses are complete, the Sample Coordinator files the form in the COC project file.

### 7.3.5 Work Sheets/Project Sheets

Work sheets are the analytical assignment forms generated by the computer or the lab manager within 24 hours after log-in for each project or group of projects. The work sheets are divided into work stations, i.e. the analytes for which one or more analysts has sole responsibility. In many cases, the work sheets will have an entry position for the results of each analyses for each sample. In either case, the work sheet, upon completion of all analyses, will be turned into the appropriate supervisor with the proper bench sheets attached. Unless specifically advised, data will not be accepted on any form other than the project approval form sheets.

### 7.3.6 Bench Sheets

The analysis of every analyte or group of analytes needed, i.e. VOA's requires a specific bench sheet which includes all results from the analysis of a group of samples. There are specific bench sheets for each analyte including specific requirements for their use. Examples of each bench sheet, can be found in Figures 8, 9 and 10.

### 7.3.7 Lab Notebooks

The lab notebooks are the daily records of all activities of an analyst, or group of analysts, working in the lab. The notebooks will be bound and paginated. The notebook will be cleanly labeled on the inside cover with the date issued, the analyst's name, and the date completed. There are several specific rules which will be follows:

- o All entries are in ink
- o There are no erasures, obliterations, or white outs allowed

- o Corrections are single lined and initialed
- o A new page is started each day or with every batch of samples
- o Empty space is covered with a Z and signed and dated across the obtuse line
- o Any and all work, observations and errors are noted
- o Problem areas identified

When the instrument has just been repaired, a lamp changed, new column installed, detector repaired, or changed in any other manner, the log will also contain:

- o A comment relative to the change or repair
- o Reference page number to the Instrument Maintenance Log

The organic log books will also contain the following information relative to GC and GCMS oven and column conditions UNLESS they are exactly as specified in the referenced method which then will be commented on as such:

- o column used (packing, diameter, length, type)
- o capillary as split or splitless
- o current type and flow
- o make up flow if appropriate
- o oven temperature and program if appropriate
- o injector temperature

- o detector temperature

- o ion chamber voltage (GCMS)

### 7.3.9 Instrument Maintenance Log

The instrument maintenance log is a bound and paginated log which is used to track potential maintenance problems. The log is used every time the instrument is used but may contain several entries on one page. Entries on days where calibrations are correct may be as simple as "calibration met requirements". Anytime the instrument is repaired or modified in any way, the event must be noted with all specifics, including what was done, by whom, and why. A two detector GC has one log tracking, two detectors.

### 7.3.10 Oven, Refrigerator and Freezer Temperature Logs

Each oven, insulator or furnace, plus all cold storage devices, will have their temperatures checked and recorded daily, or at a minimum, 5 days a week. Each device will have a thermometer in place or a temperature recorder in-place which will be checked by the Data Coordinator. A bound log book with 31 entries will be used to record all entries for each device upon which the DC will record the date and temperature and will initial the entry. The DC will have an NBS thermometer which will move between devices to act as a QC check for the primary temperature device. The log will include the second temperature when measured monthly.

### 7.3.11 Balance Logs

An Area Analyst will check all balances in the laboratory every day (or at least 5 days a week) using NBS class S weights. The analyst

will record each day's reading in a log developed to handle every balance. A balance which fails to meet criteria will be removed from service until repaired. The DC will insure that every balance is serviced and calibrated annually recording such service in the log.

### 7.3.12 Standard Record Books

Every standard used in the laboratory must be labeled and the label will possess the following information:

- The analyte or analytes contained in the standard
- The concentration
- The solvent
- The preservative, i.e. nitric acid
- The date made
- The Standard Reference Number

The last item, Standard Reference Number, is the identified standard and dilution sequence no. taken from the Standard Record Book in which the standard solution data is recorded.

All standards (including dilutions) will be recorded in a Standard Record Book assigned to the work station. Two record books will be used, each of which has a different purpose. The record books are subtitled as follows:

#### 7.3.12.1 Stock Standards Log

This book contains standards starting with the identification of the starting material. One standard and/or standard mix with it's corresponding dilutions are identified.

#### 7.3.12.2 Working Standards Log

A working standard reference number is assigned and the corresponding dilutions are identified.

#### 7.3.13 Control Charts

Each analytical method will require at least one control chart. Some tests may involve several control charts, i.e. duplicate, matrix spikes and method spikes. The QC coordinator will supply the limits to be used to the work station involved. Every data point generated with every analytical batch will be plotted on the chart. Every out-of-control data point will be noted and an action indicated as to the disposition of the data. Completed control charts will be turned in to the DC for permanent change.

#### 7.3.14 Preliminary Reports

After all data has been entered for a project, the computer will flag a project ready for a preliminary report. The report will be identical to the final report in content except for the following:

- o Preliminary Report will be reviewed and corrected if necessary on each page in large type.
- o Comments necessary to the project will be printed under each sample or at the end of the report.

The DC will print the preliminary report and issue a copy along with the project file to the lab supervisor for review and corrections. The supervisor will sign off on the preliminary report after including comments, if appropriate, indicating that corrections are necessary. Afterwards, the supervisor(s) will pass the preliminary report to the QC Supervisor (QC) who will review and correct the report including a signature and comment. The QC will return the preliminary report and file to the DC. The DC will make all corrections as required and review report structure for completeness. If no corrections are required, the DC will sign and date the preliminary report and place it in the Project File. The DC will then print a Final Report. When corrections are necessary, the DC will execute all corrections and indicate such changes on the initial preliminary, which is then filed in the project file. A new preliminary is then printed and issued for review.

#### 7.3.15 Final Report

After the preliminary report has been corrected and cleared all reviews, the DC will manually alter the computer flag and print a Final Report which will be placed in the project file folder and forwarded to the AM for approval. Space will be provided on the c.a.c. project file folder for the signatures of the Analytical Manager, the Corporate QC Manager and the Project Manager, all of whom are thus certifying that the report is complete, correct and defensible. The DC will then arrange for delivery of the final report.

### 7.3.16 Project Files

The Project File is the comprehensive record of every project completed at EDI. A project file initially consists of a file folder set up by the Lab Secretary (LC) at the time of log-in. Chain-of-Custody projects will be stored in a locked COC file with strict limited access while routine project files are stored in a separate nominally limited access file. The LS will be responsible for including the following in the project file:

- Project Sheets
- Project Approval Sheets (if applicable)
- Problem Project Sheets
- Chain-of-Custody Forms (3 each)
- All correspondence or documents received with the samples

The Lab Secretary (LS) will be responsible for the inclusion of the following:

- Preliminary Reports
- Separate Report Papers, i.e. Field Reports (if applicable)
- Final Report
- Any additional paperwork which may follow the report

All project files are stored for a period of 4 years.

## 8.0 INTRODUCTION

All samples received at the EDI Engineering and Sciences must be logged in before any work is performed on the samples. This procedural requirement is specific not only to the chemistry lab, but the micorbiological laboratory. The purpose of the log-in procedure, including sequential numbers assigned to all samples received in the facility, is to insure that EDI has a means by which samples can be tracked, data can be stored, and quality control can be tracked for any sequence of events during a particular analytical period. In handling projects in this manner, EDI, or the client, can insure a consistent and documented sequence of events under any analytical situation.

Management acknowledges that there are situations in which log-in of samples will be difficult due to rapid turn around requirements for particular compounds that may decompose or volatilize. An example of this kind of analysis is the total coliform samples which can be anticipated and for which holding times are short. The project approval form discussed within this manual will make it possible to preassign project numbers to samples arriving at the facility. Should a secondary mode of operation be necessary for the receipt of such samples, a mechanism will be developed between the sample coordinator and the Quality Assurance Supervisor. Any deviation from the standard log-in procedures detailed herein will be at the discretion of the laboratory supervisor or the laboratory manager. The execution of the log-in procedures for Chain-of-Custody samples (see Section 8.8) is extremely crucial. Samples, that have been designated for Chain-of-Custody by a client, possess the potential of involvement in litigation or other legal situations., i.e. standards development or patents. By breaking Chain-of-Custody requirements, all results are invalid for such purposes.

## 8.1 PROJECT INFORMATION

All information relative to a specific project must be recorded on a project approved form by the manager responsible for that project prior to

the receipt and log-in of samples. Projects, and therefore samples which are not routine to the EDI laboratory, must have prior approval via the New Project Approval Form before samples may be received.

## 8.2 NEW PROJECT APPROVAL

The project approval form include the following information:

- \* Client name, address, and client contact personnel
- \* Anticipated due date of the report (i.e. report in client hands by \_\_\_\_\_)
- \* Compound names or computer test codes or group computer test code
- \* Project and sample comments
- \* Contract number or purchase order for project
- \* Instructions relative to the proper completion of the project
- \* Pricing information relative to the proper completion of the project
- \* Chain-of-custody requirements
- \* Specific report requirements
- \* Additional requirements such as rush, hazardous, labile

## 8.3 NEW PROJECT APPROVAL

If a new project will require support from the analytical facilities, that project must be approved by the laboratory supervisors and the laboratory manager prior to project pricing and sample receipt. Routine samples are those samples and analyses which are continuously processed by EDI, such as priority pollutant samples, microbiological samples, and drinking water samples.

Projects which are non-routine are those that may require special testing, or which request parameters not routinely run within the laboratory, special holding times, or rush turn around. Non-routine projects will require that a New Project Approval Form be completed which includes the signatures of all the parties involved with the project. For example, if specific physical testing is necessary, the supervisor of the physical testing facility and the laboratory manager will have to sign off

on the form thereby agreeing, not only to the project content, but for the turn around, the report requirements, the detection limits and the quality control reports that may be necessary to properly carry out the project requirements. Projects and/or samples arriving at EDI which are non-routine in nature, and for which there is no signed Project Approval Form, will not be processed. In this case, the manager responsible for the non-routine project will be advised of the problem and will then explain to the client why the delay is necessary for the execution of testing before proceeding to obtain the necessary approvals. The Project Approval Form must be completed and signed by all parties prior to the start of log-in.

## 8.4 SAMPLE RECEIPT

### 8.4.1 Introduction

All samples will be received at the EDI facilities by the Sample Coordinator (SC). The job description for the Sample Coordinator is attached as Figure 11. It will be the responsibility of the SC to determine: a) whether or not the proper project sheet is available for the arriving samples; b) whether or not the samples require chain of custody; c) whether or not the samples are labile in nature and require immediate attention; d) the manner in which those samples will be split, preserved and stored or routed. It is the objective of the SC to insure that the receipt of all samples is consistent with the requirements of the EDI Manual and that all pertinent information relative to those samples is recorded. This information may be used in client reports, communicated to the laboratory or to the client and, in some cases, reported to a legal authority relative to Chain-of-Custody samples.

### 8.4.2 Examination of Shipping Container

Immediately upon receipt of a sample shipment at EDI, the SC will examine the shipping container (the container may be a box, a cooler, a styrofoam container, etc.) to ascertain and document the

condition of the samples and to process Chain-of-Custody papers, where appropriate. The SC will record the condition of the shipping container, the identification of the shipper, the presence or absence of any seals on the container (if it is Chain-of-Custody), and the labeling which may include special instructions prior to opening the container. If the shipping container is damaged, a report will be sent immediately to the shipper and the lab supervisor (see Section 8.15.2, Problem Project Sheet).

#### 8.4.3 Carrier Sign Off for Chain-of-Custody Container

Should the SC identify the shipping container as being a Chain-of-Custody container, the SC will attempt to have the carrier's representative sign off on the Chain-of-Custody papers which should be available either on the outside of the shipping container, or immediately inside. An example of a Chain-of-Custody record is attached as Figure 5. In the event that the carrier's representative is unwilling to cooperate in this fashion, the SC will identify, in the proper position on the Chain-of-Custody document, the shipment number, the date of receipt, and sign off, attaching a copy of the shipping log for that particular container.

### 8.5 EXAMINATION OF CONTAINER CONTENTS

Unless the shipping container contents are marked "hazardous" the SC will proceed to open the sample container. If the SC had not previously identified the project sheet appropriate for these samples, the SC will attempt to ascertain immediately the origin of the samples found in this container and obtain the appropriate project sheet. If a project sheet is not found, the SC will lock up the samples and notify the lab manager as described in Section 2.0. The SC will identify whether or not all the samples have arrived intact, whether or not the labels are intact and attached properly, and whether or not the samples have leaked in any fashion. The SC will also identify any shipping instructions, field instructions, or any other materials that may be present in the shipping container.

### 8.5.1 Chain-of-Custody Shipments

Should the SC identify the shipping container as a Chain-of-Custody project, the SC will immediately follow the procedure outlined in Section 4.0, "Chain-of-Custody Samples".

## 8.6 PROJECT VERIFICATION

The sample coordinator, having opened the shipping container and examined all the samples, will verify that the project sheet matches the samples, the number of samples received is consistent with the project sheet, and that the requirements identified on the project sheet are consistent with any paperwork obtained which will include the project sheet and any other documents in the sample container. The project files will be kept by the SC in a locked filing cabinet. If all required project information is not complete, the SC will fill out a Problem Project Sheet (see Section 5.2) and turn it over to the Project Manager.

## 8.7 LABILE SAMPLE DISTRIBUTION

Should the SC identify labile samples within the shipping container, (i.e. coliforms or nitrites) for which there is a very short holding time and a need to rapidly move the samples into the laboratory, the SC will make every effort to immediately log-in those samples. Should log-in be delayed, the SC will coordinate with the responsible analytical group in order to move the samples into analysis. The coordinated effort will include means by which the SC can label the samples after log-in and insure that the results correlate with the proper samples. The SC will provide computer generated sample identification to the responsible analytical group. It will be the responsibility of the SC, once labile samples have been distributed to the laboratory to insure that those samples are properly logged in and that they are labeled with properly sequenced numbers. The agreement that is made between the SC and the appropriate laboratory manager or laboratory supervisor will be based on the premise that the SC understands that he/she is ultimately responsible and will be held accountable for any samples that are lost in such a

movement. Consequently, the SC will find the samples that are labile and apply the necessary labels.

If a shipping container is labeled "Hazardous", the SC will immediately notify the laboratory supervisor who will determine the extent of hazard and/or the manner in which the samples will be handled. The supervisor will involve the laboratory manager as needed in resolving questions of hazardous samples.

## Figure 11

### POSITION DESCRIPTION FOR SAMPLE COORDINATOR

#### General

The Sample Coordinator (SC) is responsible for the receipt, log-in, and storage of all client samples at EDI. The SC is responsible for the receipt, storage and custody of all Chain-of-Custody (COC) samples including distribution of COC samples to lab personnel per EDI COC procedures (section 4.0, EDI Log-in Procedure). In order to ensure the successful analyses of samples, it is critical that the SC obtain and communicate to Project Manager, lab supervisors, and lab personnel, all information necessary for the processing interpretation and reporting of all samples analyzed.

#### Qualifications

High School Diploma and a minimum of 2 years of college or equivalent. A knowledge of chemistry and testing procedures helpful. Excellent verbal, written and organization skills, including a propensity for detail necessary for successful completion of job.

#### Reporting Relationships

The SC will report to the laboratory manager. The SC will communicate closely with the Director and Project Managers to obtain project information.

## Specific Responsibilities

The SC's duties and responsibilities shall include, but not be limited to:

1. Sample receipt.
2. Insuring that COC sample receipt includes shipper's signature on COC forms.
3. Inspection of sample shipping containers for presence/absence and condition of:
  - a) custody seals, locks, "evidence tape", etc.
  - b) container breakage and/or container integrity
4. Recording conditions of both shipping containers and sample containers (bottles, jars, cans, etc.) in appropriate logbooks or on appropriate forms.
5. Signing appropriate documents shipped with samples (i.e., Chain-of-Custody record(s)).
6. Verifying and recording agreement or non-agreement of information on sample documents (i.e., separate tags, Chain-of-Custody records, traffic reports, airbills, etc.) on appropriate forms and on the EDI project sheet.
7. Initiating the sample and project log-in procedures on appropriate laboratory documents and according to the EDI Log-in Procedures document, including the initiation of project files with sample control records.
8. Marking or labeling samples with laboratory sample numbers, as appropriate.
9. Placing samples and spent samples into appropriate storage and/or secure areas.
10. Controlling access to samples in storage and assuring that laboratory operating procedures are followed when samples are removed from and returned to storage.
11. Monitoring storage conditions for proper sample preservation such as refrigeration temperature and prevention of cross-contamination.
12. Returning shipping containers to the proper client or licensed disposal facility.

13. Providing for the splitting of samples into required aliquots, including preservation for each working station.

## 8.8 CHAIN-OF-CUSTODY SAMPLES

### 8.8.1 Continuance of Log-In Procedures for Chain-of-Custody Samples

All samples in the possession of EDI under Chain-of-Custody (COC) procedures must be traceable from the time the samples are received at the EDI door (or collected by EDI staff) until results are reported and sample disposition has been determined from the client. For any samples that may be collected during enforcement investigations, under litigatory requirements, or evidentiary in nature, Chain-of-Custody procedures are required.

### 8.8.2 Examination of Container Contents

Although Section 8.4.2 under Sample Receipt discusses the thorough examination of container contents, the proper examination of a container which is involved in a Chain-of-Custody procedure is even more important. For example, should the sample labels be mismarked or a particular sample to somewhat strange in nature, it is necessary to note all observations and deviations from the project sheet. It is better to be overly observant than to allow possible anomalies to go unnoticed. It is the SC's responsibility to examine whether or not each of the sample containers are individually sealed, whether those seals are intact, whether a sampler's initials are on the seals, and whether or not the paperwork matches the contents of the package. In addition, the SC must note whether or not all the dates and times are consistent, and whether or not the sample description on the paper work matches the description on the sample container.

## 8.9 PROJECT VERIFICATION

In the same manner in which the examination of the container contents is critical to a COC project, the verification of the project is equally important. These project verification steps include not only the need to follow the requirements identified in Section 8.6, but also thorough examination of all aspects of the project and the consistency of all the paper work involved with those particular samples in that shipping container. It is also important that the SC place in the COC project file: the shipping document; a signed Chain-of-Custody document including the sign off from the shipper's representative (See Section 8.4.3); a copy of the project sheet; a copy of the Project Approval Form is appropriate; a copy of the filed sampling report if appropriate; and originals of all paperwork received for the project. The COC project file is kept in locked storage in the possession of the SC.

## 8.10 CHAIN-OF-CUSTODY LOG-IN

The log-in procedure identified in section 8.15 titled "Log-in", is followed in the same manner for Chain-of-Custody samples with a few modifications. Those areas which are changed are addressed in the following sections:

- SAMPLE STORAGE
- PROJECT FILES
- LABORATORY ACCESS
- DATA STORAGE

## 8.11 CHAIN-OF-CUSTODY SAMPLE STORAGE

All samples received under Chain-of-Custody procedures will be kept under locked storage and will be distributed for analysis to the laboratory only when the analyst has signed for the samples on the form shown in Figure 7. The SC or a designated representative will provide access to COC storage. Records of movement of all COC samples within the lab facility must be recorded.

## 8.12 CHAIN-OF-CUSTODY PROJECT FILES

All Chain-of-Custody project files will be kept in a project folder in a locked cabinet with all related documents and paperwork relative to those files.

## 8.13 MAINTENANCE OF LAB CUSTODY

Laboratory custody must be consistent with all the Chain-of-Custody requirements from the beginning of sampling to the final report. To this end, every analyst requiring access to the Chain-of-Custody samples will go to the SC for access to the COC locked sample storage. The SC will insure that the analyst signs for the receipt of all COC samples on the form shown in Figure 7 and that the analyst returns and signs in those same samples on the same day for which they were signed out. This documentation, after the completion of all analyses, will be placed in the locked Chain-of-Custody project file by the SC.

### 8.13.1 Sample Custodian

The COC sample custodian at EDI will be designated as the Sample Coordinator (SC). The SC is responsible for following the COC requirements outlined in these procedures for all samples received at EDI.

### 8.13.2 Lab Custodial Responsibilities

It will be the responsibility of every analyst signing for a Chain-of-Custody sample or samples to insure that; a) these samples are kept in a minimum access facility; b) they are within their possession during the particular period during which they are being analyzed; and c) the analyst returns those samples to the Chain-of-Custody lock-up in the manner prescribed. The analyst will sign out and return the samples to COC lock-up on the same day. The analyst will be using the SC as the sample custodian for all COC samples. Due to the legal implications for the client of breaking the COC procedures and possibility of legal action that could be

taken against EDI, errors in the execution of Chain-of-Custody procedures will not be tolerated.

#### 8.14 CHAIN-OF-CUSTODY SAMPLE DISPOSAL

All samples received for COC procedures will be stored in the EDI COC lock-up facilities until a final report is issued. It will be the responsibility of the Project Manager, in cooperation with the SC, to obtain information from the client relative to the length of time the COC samples will be stored. It is anticipated that for long term storage, i.e. more than 30 days, the client will reimburse EDI in an appropriate rate for keeping completed samples under Chain-of-Custody procedures. No Chain-of-Custody samples may be discarded until written permission is received from the client relative to disposal of those samples.

#### 8.15 LOG-IN

##### 8.15.1 Introduction

After the SC has inspected the shipping containers, the project sheets, the samples and any documentation required in Sections 8.4 and 8.8, the SC will insure that all pertinent information is entered on the project sheet. There are specific areas of the project sheet that are to be completed by the SC, i.e., date and time received. The EDI project sheet is included as Figure 2.

Minimum information required for log-in include:

- \* Client's name and Client contact, as well as client #, is assigned.
- \* The due date
- \* The analytical test or test codes or group tests
- \* Specific project comments
- \* Contract requirements
- \* Contract number

- \* Pricing if necessary
- \* The approval for for non-routine projects
- \* Chain-of-Custody, if required
- \* Specific report requirements

#### 8.15.2 Project Problems

If any of the information identified in sub-section 8.15.1 is missing, the SC will immediately notify the Project Manager, via a Problem Project Sheet, (Figure 4) of the discrepancy. The Project Manager will make all reasonable efforts to insure that the answers are provided to the SC immediately.

Simple Project Sheet deficiencies such as client number, extra comments, or the contract number, should not prevent log-in. The SC will proceed with log-in addressing the unknowns as subjects that must be changed or modified once the information is received. It is the responsibility of the SC to log-in all samples as received at EDI whenever possible.

#### 8.15.3 Samples on Hold

When there is a considerable amount of inadequate information on a project sheet, i.e. a missing test, or broken samples, the entire project will be placed on hold until the information is available or the corrective actions have been taken to insure that NSF is not held responsible for a poorly handled project. The SC will notify the Project Manager via a Problem Project Sheet as to the hold status of the project and the reasons for the hold. The Project Manager will make every attempt to quickly identify the necessary actions that will be taken for those samples or the remaining samples for that project. The Project Manager may approve log-in of the remaining samples for a portion of the project in order to insure that the project progresses. Projects that are placed on hold will be locked in a "project hold" area, (like the Chain-of-Custody sample storage area) so that those samples are not lost or confused within the system. The SC will insure that those

samples are retrieved and logged in as soon as the appropriate changes have been made and the samples are freed for log-in.

#### 8.15.4 Handling Labile Samples

All samples received by the SC that are labile in nature, i.e. coliforms, need to be logged into the facility in a very rapid fashion in order that they may be attended to within the analytical holding time. The most labile of all samples are the microbiological samples, which must be forwarded to the micro lab as soon as possible. The SC and the Project Managers responsible for micro work will attempt to insure that appropriate information is available to the SC in order that the SC can assign numbers for all labile samples. These numbers can be assigned in advance and samples may be logged into the system as soon as they are received. Samples such as nitrites, which are labile but have a somewhat longer holding time, will usually be logged into the system like normal samples. However, slow shipment or other problems may require the lab to initiate the analyses immediately. In such a case, assuming a project sheet was initiated in advance of sample receipt, the SC can assign laboratory in an expedient fashion. The SC will make all efforts to insure that samples move through the laboratory in a timely fashion when holding times are of utmost importance to the proper completion of the analytical requirements.

#### 8.16 COMPUTER LOG-IN

It is anticipated that all samples received at EDI will be logged on to the computer by the SC. The computer assigns a sequential number to every sample. Additional codes such as the month and the year of the samples may be added in front of the sequential number for continuous identification of these samples. The SC will have the computer generate these sequential numbers for each sample in every project. A project identifier will be printed on the labels which are attached to every sample and every aliquot of a sample.

## 8.17 SAMPLE SPLITTING FOR THE CHEMICAL LABORATORY

The EDI Project Manager will attempt to insure that all samples received at the EDI facility are received in the appropriate containers with the correct preservatives (Samples which must be split at log-in are subject to added error). The labels and the appropriate preservatives are identified in the EDI Quality Control Manual and attached as a supplement to this section in Appendix I.

### 8.17.1 Bottles and Preservative Requirements

The EDI analytical facility has a series of bottle and preservative requirements that must be met before the log-in of samples into the laboratory. In the event that EDI is unable to provide sample bottles, or circumstances prevent the splitting of samples in the field, the SC will provide sample splitting services. These services will include taking the sample as received and subsampling it into the appropriate bottle and preservative requirements as set forward on the attached list of bottle and preservative requirements.

### 8.17.2 Inorganic Samples

The SC will insure that sufficient sample volume is available before initiating the splitting of a sample. If uncertain, the SC will involve the inorganic lab lead in order to insure that all areas of the lab have sufficient samples. In the event that sufficient samples does not exist, the SC will identify the sample as a problem and will notify the Project Manager immediately for resolution. The sample will be logged in only after a resolution has been reached.

### 8.17.3 Organic Analysis

When a bulk sample arrives for organic/inorganic analysis and sufficient sample exists, the SC will transfer the sample to the

organic preparation supervisor who will split the organic aliquots and return all aliquots to the SC. The remaining sample will then be returned to the SC who will split off the inorganic aliquots into the proper preserved containers.

#### 8.17.4 Solid Samples Splitting

When solid samples, such as sediment or soil, are to be received at EDI, every attempt will be made by the Project Manager and field sampling personnel to insure that two samples are provided as replicates for the appropriate tests. One of these samples will be assigned to the organic facility; the other will be assigned to the inorganics facility. If only one sample is received and if organic analyses are required, the organics preparation chemist will be responsible for the initial splitting of the sample. Solid samples will be made homogenous by either one or all of the following manners:

- \* Stirring especially when volatile organic analytes are required
- \* Air Drying and Grinding
- \* Particle separation (Sieving)
- \* Quartering by ASTM Procedures

The lead organic chemist and the SC are responsible for the decisions on how a solid sample will be split. Problems or concerns which may arise on a solid sample will be addressed to the Project Manager and the laboratory manager for resolution. After the organic portions have been removed or split, the remaining sample will be provided to the inorganic facilities for any further splitting they deem necessary.

#### 8.18 SAMPLE LABELING

All samples received at the EDI facility will be labeled by the SC at the time of log-in. These labels will include information such as the requested sample number, the client number if supplied, the contract, the

EDI project number, and/or the client. It is anticipated that sequential sample labels will be provided by the computer after the SC has logged the project into the computer.

## 8.19 DISTRIBUTION AND STORAGE

Logged samples will be taken by the SC to the appropriate walk-in cooler for cold storage or to the room temperature storage area indicated for metals.

COC samples are stored as set forth in Section 4.0.

## 8.20 PROJECT FILES

### 8.20.1 Routine Project Files

The SC will obtain a manila folder and label that manila folder with the name and number of the project. The folder will indicate the EDI project number, the EDI contract number, and Chain-of-Custody if applicable. With the agreement of the laboratory supervisor (lead), the project manager, and the laboratory manager, a particular project folder may include a series of projects logged in under sequential numbers. An example would be a daily log-in for the same project for a week or month before a new project folder is generated. It is, however, the responsibility of the SC to insure that all logged projects are filled in a project file folder.

### 8.20.2 Chain-of-Custody File Folder

The SC, upon logging in any Chain-of-Custody project, will provide the same type of manila folder project file, as discussed in Section 5.7.1, for each project. However, the project folder will be maintained in the locked Chain-of-Custody file and cabinet and will be kept by the sample coordinator.

## 8.21 SAMPLE STORAGE

### 8.21.2 Non Chain-of-Custody Storage

The SC, after completing all the log-in processes of various samples connected with a particular project, will store the samples in the designated areas in the EDI laboratory.

- \* Routine Water and Solid Samples: Samples which need to be refrigerated will be stored in the walk in facility designated for all routine water and soil samples.
- \* Routine Volatile Water and Solid Samples: All these samples will be placed in the designated VOA refrigerator located within the analytical facility. No other samples or standards may be stored in the VOA refrigerator.
- \* Routine Water and Solid Samples for Metal Parameters: The preserved water samples and solid samples, which are not preserved, may be stored on shelves designated for the metals analysis.
- \* Odoriferous and Hazardous Samples: These samples will be stored in a hooded facility within the laboratory which is designated for odoriferous and hazardous samples. These samples will be identified to the lab personnel and noted on the log-in procedures in order to insure that the lab personnel are aware of the problems with these samples.

## 8.22 CHAIN-OF-CUSTODY SAMPLE STORAGE

All samples that are involved as physical evidence in a legal procedure or simply identified as Chain-of-Custody will be handled under certain procedural safeguards. These safeguards have been tentatively identified in section 4.0 but for purposes or reiteration are again addressed below:

NOTE: For any legal proceedings, the court must be shown that the laboratory is a secured area, that all samples have been stored in a secured fashion, and samples can be accounted for at all times.

#### 8.22.1 Chain-of-Custody Water and Solid Samples

All samples of this nature will be stored within the locked cabinets in the designated walk-in cooler for routine samples. Keyed access is available only through the SC.

All samples received for metals analysis under Chain-of-Custody procedures, whether they be liquid or solids(s), will be stored in a locked cabinet which does not need to be in the walk-in facility. Such a cabinet will be designated and placed by the inorganic lab supervisor.

#### 8.22.2 Water and Soil Samples for Metals

### 8.23 GENERAL LAB SECURITY

Access to the EDI lab will be handled in a secured fashion restricting entrance only to those people designated as having access to the laboratory facilities. Restricted access applies to all areas in which samples are stored or analysis takes place. It will be the responsibility of all the analysts, as well as the supervisors and the SC, to insure that the safeguards employed, including locked doors and limited access, are followed and maintained at all times.

## 9.0 DATA HANDLING, REPORTING, RECORDKEEPING AND VALIDATION

There are two significant aspects of any analytical procedure:

- A. The selection and use of a method appropriate for the analyte and matrix
- B. The collection, control and interpretation of the data generated.

Encompassing these two components is the Quality Assurance program. The QA program provides means by which method selection can be validated, the method can be controlled and the appropriate data generated, displayed and reduced.

The following sections deal with error, data handling, data validation, data reporting and data recordkeeping.

### 9.1 ERROR: IT'S NATURE AND SIMPLE STATISTICAL CONCEPTS

#### 9.1.1 Random Errors

Repeated analysis of identical aliquots of a homogeneous sample does not give a series of equivalent results. The results will differ among themselves and they will be more or less scattered about some value. The scatter can be attributed to random error, so named because the prediction of the sign or magnitude of the error of any particular result is not possible at the time of analysis.

One therefore, says that each result must have an uncertainty attached to it, and can be regarded only as an estimate of the true value. Generally that estimate will differ from the true value. Random errors are caused by uncontrolled and/or uncontrollable random variations in factors which affect analytical results, i.e.

variations in the volumes of the reagents added, variations in the concentrations of reagents, variations in the time allotted for the chemical analysis, a contaminated glassware, poor quality reagents, instrumental fluctuations. Among the various texts that are available discussing errors, the terms repeatability, reproducibility and precision have been used to denote the scatter of results. The term "precision" will be used throughout this manual and is the most common term used for random error in this country and especially by the EPA.

Precision does improve as the scatter among results becomes smaller. All analytical results have random error present which necessitates statistical techniques to evaluate the results and to provide correct inferences of the true value of the result.

## 9.2 SYSTEMATIC ERRORS

Systematic errors are indicated by the tendency of results to be greater or smaller than, the true value. It is necessary to take care in exactly defining systematic error because the analysis is also subject to random error. The mean of  $n$  analytical results on the same sample approaches a definite value  $u$  as the number of results increases indefinitely. When  $u$  differs from the true value  $\tau$  results are said to be subject to systematic error of the magnitude  $B$ , wherein  $B$  is equal  $u$  minus  $\tau$ . Bias is the term used synonymously with systematic error and will be used in that fashion throughout this manual. Analytical methods, which are subject to interferences from substances present in the sample, or methods that only recover a fraction of the material present are an example of systematic error.

It is impractical to make an indefinitely large number of analysis on a single sample in order to determine the true value of  $u$  is known. At the same time a practically obtained value for a sample that is based on minimal analysis is subject to random error, so that the experimental estimates of bias will also be subject to random error. Therefore, statistical techniques are also required when bias is to be estimated.

The basic difference between random and systematic error is that, in principal, the latter may be predicted so that a correction can be made to eliminate its effect. An example of this allowance can be accounted for in the effect of fluoride in the determination of aluminum by absorbance measurements. This effect is overcome by adding to the calibration standards an amount of fluoride equal to the fluoride content of the sample. The added fluoride in the calibration standards then eliminates the systematic error of fluoride interference. However, it must be recognized that the complete elimination of systematic error may require such detailed knowledge of the properties of the sample that the correction of the analytical system is impractical and would in fact increase the amount of random error. Thus, in all applications where unbiased results are necessary, the approach to be used is to devise and use analytical systems capable of giving results which have negligible systematic error.

### 9.3 TOTAL ERROR

Some analysts use the term accuracy to denote only systematic error. The term accuracy as applied in this manual will denote total error of the results. In other words, accuracy represents the combined systematic and random error of the results and, therefore, the accuracy of an analysis improves as the total error becomes smaller. For the purposes of visually seeing random and systematic error, Figure 6-1 should be referred to for any easy identification of the various types of error.

### 9.4 STATISTICAL TECHNIQUES

Statistical techniques are essential to the measurement of analytical error. This manual and this section recognize that many analysts have had little experience with statistical technique. This section is, therefore, written in such a way as to explain simple but basic concepts of the statistical approach and to describe the particular techniques most commonly required in dealing with analytical errors. There are a large number of text books dealing with statistics and this particular section does not attempt to replace these books. The intention is merely to

present the essential aspects in the simplest manner possible. Certain approximations have been used when considered appropriate and no previous knowledge of statistics has been assumed. Should the analyst be interested in consulting additional texts for a more rigorous and detailed treatment of the subject, he is referred to the references at the end of section 9.0.

Analysts who are unfamiliar with statistical approach, may find this section on first glance rather complicated. In order to understand statistics for the QC function, it is important not to be put off by the first impression.

The fundamental statistical concepts are essentially simple and equivalent to the intuitive common sense, or perhaps scientific approach, adopted by any good analyst.

#### 9.4.1 Random Error Distribution

If the results from the analysis of numerous aliquots of a homogeneous sample are plotted on a histogram, it is generally found that the proportion of the results deviating from the mean increased, i.e., as the deviation of the results from the mean grows broader. In other words, the probability of obtaining a random error of a given size decreases as the size of the error increases. The basis of statistical techniques is to quantitatively estimate the probabilities of errors of different sizes so that one can deduce the probable random error of a particular analytical result. If the analyst were to increase the number of analysis of a single sample indefinitely, and the size of the intervals used for plotting the histogram were decreased, the latter would tend to smooth the curve. This limiting curve is the frequency distribution of results and defines a relationship between the magnitude of the result and the probability of obtaining such a value. Throughout this manual, it will be assumed that the analytical results follow the normal distribution which is defined by the following equation:

$$p(x) = \mu \pm \sigma$$

Where:  $\mu$  = the mean of all the conceptionally infinite number of results.

$\sigma$  = the standard deviation of results

$p(x)$  = the probability density which is interpreted by noting that the probability of obtaining a result between the values a & b is the area of the curve between those values.

and this interval can be evaluated given the equation for  $P(X)$ .

The peak of this distribution curve occurs at  $x=u$ , the theoretically perfect mean established by an infinite number of results. The width (which is indicated by the scatter results) is determined solely by the standard deviation of the test. For example, 95% of the area under the curve, i.e. 95% of all results, is enclosed within the limits plus or minus 1.96. Such properties allow limits for the uncertainty of an individual analytical result to be calculated. Taking the current discussion, for example, on no more than 5 occasions in one hundred will the result differ from the mean  $u$  be more than 1.96. Thus, an analyst may attach to a result limits that define the range in which the true mean is expected to lie. The statement,  $R-1.96$  is less than  $u$  which is less than  $R+1.96$ , is an accurate statement on 95% of all occasions. "R" in this particular case would stand for the result. By referring to texts on statistics, there are statistical tables which included a tabulation of areas enclosed between specific limits as an analyst might want to define them. It should be noted that the distribution is always symmetrical about the mean. In other words, if one is using the 1.96 levels 5% of the results will be outside of the range of  $u \pm 1.96$ , but only 2.5% of all results will exceed  $u + 1.96$  and 2.5% of the results will be less than  $u - 1.96$ .

Focusing this into a discussion more pertinent to the laboratory and, perhaps more viable with respect to occurrences within the laboratory, let us discuss the rare exception in which an analyst is taking 20 tests on a particular sample using the 1.96 level. Considering that 5% of the results will lie outside that level, the analyst has 1 chance in 20 of missing the true value outside the stated confidence range. At the same time one can decrease this chance by increasing the allowable range. For instance, if the range is  $R = \pm 2.58$  the results will be included on 99% of the occasions or 99% of the tests. However, by increasing the confidence limit, one is also increasing the uncertainty in the true value. In this case, uncertainty can be decreased by taking the mean of several analytical results or by decreasing the value.

These statistical concepts allow valuable quantification of the random error of an analytical result and emphasize that decisions, based on the significance of the result, have some risk of being wrong. Knowledge of the standard deviation, of the results is, therefore, vital in reaching objective decisions. Use of the standard deviation will be explained in the following sections dealing with data handling and validation.

#### 9.4.2 Data Handling, Reporting, Recordkeeping

A flow diagram, Figure 1, delineates the original and procedural steps in data generation.

The initiation of an analysis starts with the completion of a project approval form. The information is computer entered. The computer entry internally creates a report form and inventories the analysis by parameter or compound. The computer entry function of all analytical work requests is a shared responsibility of the sample coordinator and data coordinator. A copy of the analysis request form is manually inserted into a three ring binder notebook

for laboratory reference use. The maintenance of the laboratory job reference notebook is a responsibility of the sample coordinator. The group leader/supervisors requests from the data coordinator (D.C.), the computer generated analytical bench sheets for a given parameter each morning or the prior day. The samples and parameters testing sequence is dictated by a weekly work schedule. The weekly work schedule is developed manually each week by the group leaders/area supervisors and approved each week by the laboratory manager. The schedule is developed from a computer printout that inventories and ages by project job or parameter. Contractual due dates and sample holding times are the compliance criteria by which all schedules are judged.

The bench sheets examples are shown in Figure 8, 9, 10,. The bench sheets identify to an analyst the proper samples to analyze that day. The analyst lab notebook and the bench sheets constitute the two raw data reporting locations. The content of the laboratory notebook is defined in an earlier section, 7.3.7. The analyst completes the benchsheet information, attaches a drawn calibration curve and follows the analytical sample sequence identified in section 10.0. The analyst identifies which sample(s) were utilized for precision and accuracy determinations. The analyst will assess the data set as being in control or not. The assessment will be described in the data validation section to follow. The analyst will submit to respective group leaders or supervisors all of the abovementioned data and a written statement that the data set is in control for their review. An approved data set is signed off and the group leaders/supervisors transfer the approved data to all appropriate worksheets in the laboratory job reference notebook. The bench sheets and calibration curves are permanently stored. The last entry into the worksheet constitutes a completed project subject to computer generation of a preliminary report. The group leader/supervisors provide the DC with the approved worksheets for computer entry and preliminary report generation. The remaining activities related to preliminary report, final report generation and review and project filing are identified in this manual under sections 7.3.14, 7.3.15 and 7.3.16 respectively.

## 9.5 DATA VALIDATION

The data validation process includes a set of computerized and manual checks at various appropriate levels of the measurement process.

The data validation process starts with the laboratory analyst. The analyst verify in their lab notebook that all method specific operational parameters are utilized or met. This information is specifically documented in all instrument logbooks. The analyst then verifies that the calibration of the equipment is linear and documents this in the instrument logbooks. If the operating parameters of a particular method are modified, it should be written in the analyst lab notebook and approved via signature by the group leader/supervisor in the lab notebook. A non-calibrated system must be identified by the analyst and corrections made to achieve calibration prior to sample analysis.

The generation of sample data by an analyst will include the generation of quality control data for each sample set. The monitoring of method blanks, sample spikes, method spikes and sample duplicate analysis is accomplished by the utilization of Schwart Quality Control Charts. All quality control data is entered on the precision and accuracy data summary form, Figure 11a. The analyst computes the data precision and accuracy and compares the computed value to the acceptance intervals identifies on the form for that parameter, method, and matrix. The computed value will be determined in control if it lies within the acceptance interval. If the computed value is deemed out-of-control the data set is not submitted for supervisor approval but is brought immediately to the attention of the supervisor and quality assurance officer that an out-of-control condition exists. Jointly, a review is conducted to determine the cause(s) and conduct corrective action. The data set is rerun once the corrective actions have taken place and the new data reviewed as stated above.

The DC receives all the completed precision and accuracy data summary forms and enters the data into the laboratory quality control computer system. The system produces summary reports each day of all quality control data generated for review by the quality assurance officer. The

computer system also generates all Schwart Control Charts for method blanks, method spikes, sample duplicates and sample spikes. The charts are permanently maintained and reviewed each week by the group leader/-supervisor and the quality assurance officer. The weekly generated charts provide an accurate review of all recently (last 30) qc data points and allows the monitoring of data trends or other anomalies to the system.

## 10.0 GENERAL QUALITY CONTROL PRACTICES

The Quality Assurance/Quality Control practices at EDI are based on several of the following government guidelines:

- o "Handbook for Analytical Quality Control in Water and Wastewater Laboratories" EPA 600/4-79-019, March 3, 1979
- o The Guidelines Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act (Federal Register, Dec. 3, 1979, updated on October 26, 1984.)
- o Manual of Analytical Methods for the Analysis of Pesticides in Humans and Environmental Samples" EPA 600/8-80-038 June 1980.
- o ASTM

The quality control procedures used during analysis are described below and do conform with sample control and document control requirements described for the EPA Contract Lab Program (CLP). The specific sample and document control practices employed at EDI may be found in the appropriate sections of this manual identified as Sample Control (section 8.0) and Document Control (section 7.0).

The procedures described here will deal with the analytical quality control techniques. The quality control checks routinely performed during sample analysis include method blank analysis to identify potential method interferences, reagent blank analysis to establish analyte levels, duplicate analysis to establish analytical precision, spiked and blank analysis to determine analytical accuracy. The frequency of these quality assurance checks are defined in the EPA CLP and result in the following analytical run sequence:

Method Blank  
Reagent Blank  
Standard

Sample #1  
#2  
#3  
#4  
#5  
#6

Spiked Blank

Sample #7  
#8  
#9  
#10

Sample Duplicate #10  
Sample #11  
Sample Spiked #11  
Sample #12

Any high level concentrations of analyte will be followed by a method blank. The exact analytical sequence is left to the discretion of the analyst.

10.1 The level of laboratory quality assurance effort for routine analytical services (RAS) provided by EDI is specified in Table 1. The quality assurance level of effort for the Contract Lab Program (CLP) is specified in the protocol document and will be complied with.

## 10.2 ACCURACY, PRECISION AND SENSITIVITY OF ANALYSES

The fundamental QA objective with impacting accuracy, precision and sensitivity of laboratory analytical data is to achieve the QC acceptance criteria of the analytical protocols. The accuracy and precision requirements for routine analytical services (RAS) for organic and inorganics are shown in Tables 2 and 3, respectively. These QC control limits should be completely met without any outliers.

These limits are three (3) standard deviations from the mean and reflect control limit criteria established and utilized on Schwart Control Charts for precision and accuracy.

The standard operating procedures that would lead to an outlier being identified and the resulting corrective actions is described in section 9.0, Data Reporting, Validation and Handling. In general, if an out-of-control result occurs the analyst will identify it as such and report the occurrence to the Group Leader and/or Area Supervisor. The Group Leader and/or Area Supervisor will review the data with the analyst to identify the problem, implement a corrective action(s) and then re-analyze the sample(s). The Group Leader and/or Area Supervisor will report the out-of-control occurrence to the Quality Assurance Officer that day in writing. The corrective action(s) will be identified in the analyst notebook and in writing to the QA Supervisor. The QA Supervisor will review the Schwart Chart later that day or the next day to identify a successful corrective action via a new in-control data point for the same data set. The sensitivities required for RAS analyses are the method detection limits shown in Tables 4 and 5.

The accuracy and precision requirements for Chain-of-Custody (C.O.C.) samples from the CLP-HSL are specified in the protocols. The sensitivities required for Hazardous Substance List (HSL) analysis of organic compounds will be the EDI operational detection limits, shown in Table 6. Table 7 indicates HSL analyses for inorganic compounds as identified in the CLP. The sensitivity for these compounds is based upon the EDI operational detection limits as shown on the table.

TABLE 1

## QA LEVEL OF EFFORT FOR ROUTINE ANALYTICAL SERVICES

<u>PARAMETER</u>	<u>METHOD BLANKS</u>	<u>SPIKES OR SURROGATES</u>	<u>LAB DUPLICATES</u>	<u>REFERENCE SAMPLES</u>
base/Neutral/Acid compounds	One per set of samples or a minimum of 1 in 10	Surrogates added to each sample and matrix/method spikes added to one sample per set	A minimum of 1 in 15	Quarterly
volatiles	One per day or 8-hour shift	Surrogates added to each sample and matrix/method spikes added to one sample per set	A minimum of 1 in 15	Quarterly
pesticides & PCBs	One per set of samples or a minimum of 1 in 10	Matrix/method spike per set of samples or a minimum of 1 in 10	A minimum of 1 in 10	Quarterly
metals	One per 10 samples or each set	One matrix spike per 10 samples; one method spike per set	One per 10 samples	Quarterly
cyanide	One per analytical run or at least one per day	One matrix spike per 10 samples; one method spike per set	One per analytical run or at least one per set-up	Quarterly
alkalinity	One per set of samples	NA	One per set of samples or at least one per 10 samples	Quarterly
chloride	One per set of samples	One matrix spike per 10 samples; one method spike per set	One per set of samples	Quarterly
fluoride	One per set of samples	One matrix spike per 10 samples; one method spike per set	One per set of samples or at least one per 10 samples	Quarterly
sulfate	One per set of samples	One matrix spike per 10 samples; one method spike per set	One per set of samples or at least one per 10 samples	Quarterly
ammonia Nitrogen	One per set of samples	One matrix spike per 10 samples; one method spike per set	One per set of samples or at least one per 10 samples	Quarterly

Table 1 (Continued)

QA LEVEL OF EFFORT FOR ROUTINE ANALYTICAL SERVICES

<u>PARAMETER</u>	<u>METHOD BLANKS</u>	<u>SPIKES OR SURROGATES</u>	<u>LAB DUPLICATES</u>	<u>REFERENCES SAMPLES</u>
TKN	One per set of samples	One matrix spike per 10 samples; one method spike per set	One per set of samples or at least one per 10 samples	Quarterly
Nitrate and Nitrite	One per set of samples	One matrix spike per 10 samples; one method spike per set	One per set of samples or at least one per 10 samples	Quarterly
TOC	One per set of samples	One matrix spike per 10 samples; one method spike per set	One per set of samples or at least one per 10 samples	Quarterly
Total Phosphorus	One per set of samples	One matrix spike per 10 samples; one method spike per set	One per set of samples or at least one per 10 samples	Quarterly

TABLE 2

ACCURACY AND PRECISION CRITERIA FOR ORGANICS FROM RAS

<u>PARAMETER</u>	<u>AUDIT</u>	<u>COMPOUNDS</u>	<u>CONTROL LIMITS</u>
Volatiles			
Method Blank	---		d.l.
Matrix Spike Duplicate Precision	---		<17% RPD (95% CI)
Surrogate Spike Recovery		D-1,2-dichloroethane D <sub>4</sub> -benzene D <sub>6</sub> benzene Dioethylbenzene	70-115% 70-115% 70-115%
Method Spike Recovery		benzene 1,2-dichloroethane 1,1,1-trichloroethane trans-1,2-dichloroethane trans-1,2-dichloropropene cis-1,2-dichloropropene ethyl benzene toluene 1,1,2,2-tetrachloroethane bromoform	37-151% 49-155% 52-162% 49-155% 17-183% 0-227% 37-162% 47-162% 46-157% 45-170%
pesticides & PCBs			
Method Blank	---		d.l.
Sample Duplicate	---		34% RPD (95% CI at level >1 ppb)
Method Spike Recovery (normal pesticide analysis is run)		endrin lindane dieldrin aldrin 4,4-DDD 4,4-DDT endosulfan II endosulfan sulfate	69-119% 62-118% 20-136% 50-166% 10-145% 10-203% ---- 10-100%

Table 2 (Continued)

ACCURACY AND PRECISION CRITERIA FOR ORGANICS FROM RAS

PARAMETER	AUDIT	COMPOUNDS	CONTROL LIMITS
Base/Neutral/Acid Compounds	Method Blank	4,4 -DDE heptachlor chlordane	10-136% 10-192% 65-129%
	Method Blank	---	d.l.
	Surrogate Spike Recovery	---	<38% RPD (95% CI)
Method Spike Recovery	Surrogate Spike Recovery	Pentafluorophenol	10-86%
		2-fluorophenol	10-103%
		2,4,6-tribromophenol	43-105%
		D-nitrobenzene	29-107%
		2-fluorobiphenyl	25-119%
		decafluorobiphenyl	42-112%
		phenol	10-121%
		2-chlorophenol	23-134%
		1,4-dichlorobenzene	20-124%
		benzyl alcohol	10-124%
		n-nitrosodipropylamine	10-127%
		1,2,4-trichlorobenzene	22-147%
		4-chloraniline	10-100%
		p-chloro-a-cresol	10-130%
		acenaphthene	47-145%
2,4-dinitrotoluene	39-139%		
4-nitrophenol	10-132%		
pentachlorophenol	14-176%		
Di-n-butyl phthalate	1-118%		
pyrene	52-115%		
1,3-dichlorobenzene	10-172%		
2,6-dinitrotoluene	50-158%		

TABLE 3

ACCURACY AND PRECISION CRITERIA FOR INORGANICS FROM RAS

<u>PARAMETER</u>	<u>AUDIT</u>	<u>CONTROL LIMITS</u>
Metals and Other Inorganics	Method Blank	$\pm$ d.l.
	Sample Duplicates	$\pm$ d.l. or $\pm$ 10% RPD
	Method Spikes	85-115%

NOTE: RPD = relative percent difference

TABLE 4

## OPERATING DETECTION LIMITS FOR ORGANICS FROM RAS

Pesticides and PCBs

<u>CAS NO.</u>	<u>COMPOUND</u>	<u>OPERATING DETECTION LIMIT (ug/L)</u>
319-85-7	$\beta$ -BHC	0.1
319-84-6	$\alpha$ -BHC	0.1
319-86-8	$\delta$ -BHC	0.1
76-44-8	heptachlor	0.1
58-89-9	$\gamma$ -BHC (Lindane)	0.1
309-00-2	aldrin	0.1
1024-57-3	heptachlor epoxide	0.1
959-98-8	endosulfan I	0.1
72-55-9	4,4 -DDE	0.1
60-57-1	dieldrin	0.1
72-20-8	endrin	0.1
72-54-8	4,4 -DDD	0.1
33212-65-9	endosulfan II	0.1
50-29-3	4,4 -DDT	0.1
57-74-9	chlordan	0.1
8001-35-2	toxaphene	1.0
72-43-5	methoxychlor	1.0
8001-35-2	aroclor 1016	1.0
11097-69-1	aroclor 1242	1.0
11096-82-5	aroclor 1248	1.0
11104-28-2	aroclor 1254	1.0
12674-11-2	aroclor 1260	1.0

TABLE 4 (Continued)

Base/Neutral and Acid Extractable Compounds

<u>CAS NO.</u>	<u>COMPOUND</u>	<u>OPERATING DETECTION LIMIT (ug/L)</u>
62-53-3	aniline	5
111-44-4	bis (2-chloroethyl) ether	4
108-95-2	phenol	2
95-57-8	2-chlorophenol	2
541-73-1	1,3-dichlorobenzene	3
106-46-7	1,4-dichlorobenzene	3
95-50-1	1,2-dichlorobenzene	3
100-51-6	benzyl alcohol	10
118-60-1	bis (2-chloroisopropyl) ether	1
95-48-7	2-methylphenol	5
67-72-1	hexachloroethane	7
621-64-7	N-nitrosodipropylamine	4
98-95-3	nitrobenzene	4
108-39-4	4-methylphenol	5
78-59-1	isophorone	2
88-75-5	2-nitrophenol	5
105-67-9	2,4-dimethylphenol	3
11-91-1	bis (2-chloroethoxy) methane	4
120-83-2	2,4-dichlorophenol	3
120-82-1	1,2,4-trichlorobenzene	3
91-20-3	naphthalene	1
106-47-8	4-chloroaniline	5
87-68-3	hexachlorobutadiene	5
65-85-0	benzoic acid	50
91-57-6	2-methylnaphthalene	1
59-50-7	p-chloro-m-cresol	4
77-47-4	hexachlorocyclopentadiene	10
95-95-4	2,4,5-trichlorophenol	5
88-06-2	2,4,6-trichlorophenol	5
91-58-7	2-chloronaphthalene	2
208-96-8	acenaphthylene	1
131-11-3	dimethyl phthalate	2
606-20-2	2,6-dinitrotoluene	9
83-32-9	acenaphthene	2
99-09-2	3-nitroaniline	50
132-64-9	dibenzofuran	10
51-28-5	2,4-dinitrophenol	50.0
121-14-2	2,4-dinitrotoluene	10
86-73-7	fluorene	1
100-02-7	4-nitrophenol	15
7005-72-3	4-chlorophenyl phenyl ether	3
84-66-2	diethyl phthalate	2
534-52-1	4,6-dinitro-2-methylphenol	10

TABLE 4 (Continued)

<u>CAS NO.</u>	<u>COMPOUND</u>	<u>OPERATING DETECTION LIMIT (ug/L)</u>
122-66-7	1,2-diphenylhydrazine (azobenzene)	1
86-30-6	N-nitrosodiphenylamine (diphenylamine)	2
100-01-6	4-nitroaniline	50
101-55-3	4-bromophenyl phenyl ether	7
118-74-1	hexachlorobenzene	5
87-86-5	pentachlorophenol	20
85-01-8	phenanthrene	1
120-12-7	anthracene	1
84-72-2	di-n-butyl phthalate	1
206-44-0	fluoranthene	1
129-00-0	pyrene	1
85-68-7	butyl benzene phthalate	3
218-01-9	chrysene	5
56-55-3	benzo(a)anthracene	5
117-81-7	bis (2-ethylehexyl) phthalate	2
117-84-0	di-n-octyl-phthalate	2
205-99-2	benzo(b)fluoranthene	5
207-08-9	benzo(k)fluoranthene	5
50-32-8	benzo(a)pyrene	5
193-39-5	ideno(1,2,3-cd)pyrene	10
53-70-3	dibenzo(a,h)anthracene	10
191-24-3	benzo(ghi)perylene	10
92-87-5	benzidine	50
91-94-1	3,3-dichlorobenzidine	20
	octachlorocyclopentane	10

TABLE 4 (Continued)

<u>Volatile Compounds</u>		OPERATING DETECTION LIMIT (ug/L)
<u>CAS NO.</u>	<u>COMPOUND</u>	
74-87-3	chloromethane	10
74-83-9	bromomethane	10
75-01-4	vinyl chloride	10
75-00-3	chloroethane	10
75-09-2	methylene chloride	2
107-02-8	acrolein	15
67-64-1	acetone	10
107-13-1	acrylonitrile	15
75-15-0	carbendisulfide	1
75-35-4	1,1-dichloroethane	1
75-34-3	1,1-dichloroethane	2
156-60-5	trans-1,2-dichloroethene	2
67-66-3	chloroform	2
78-93-3	2-butanone	10
107-06-2	1,2-dichloroethane	2
71-55-6	1,1,1-trichloroethane	2
56-23-5	carbon tetrachloride	2
108-05-4	vinyl acetate	10
75-27-4	bromodichloromethane	2
78-87-5	1,2-dichloropropane	3
10061-02-6	trans-1,3-dichloropropane	4
79-01-6	trichloroethene	2
71-43-2	benzene	1
124-48-1	chlorodibromomethane	3
79-00-5	1,1,2-trichloroethane	3
10061-01-05	cis-1,3-dichloropropene	4
110-75-8	2-chloroethylvinyl ether	10
75-25-2	bromoform	15
108-10-1	4-methyl-2-pentanone	5
519-78-6	2-hexane	100
127-18-4	tetrachloroethene	2
79-34-5	1,1,2,2-tetrachloroethane	2
108-88-3	toluene	1
108-90-7	chlorobenzene	1
100-41-4	ethylbenzene	1
100-42-5	styrene	10
	m-xylene	10
95-47-6	o-xylene, p-xylene	10

TABLE 5

## OPERATING DETECTION LIMITS FOR INORGANICS FROM RAS

<u>PARAMETER</u>	<u>DETECTION LIMIT (ug/L)</u>
Aluminum	250
Chromium	10
Barium	250
Beryllium	10
Cobalt	10
Copper	10
Iron	10
Nickel	10
Manganese	10
Zinc	20
Boron	500
Vanadium	100
Silver	10
Arsenic	2
Antimony	100
Selenium	2
Thallium	50
Mercury	0.2
Tin	500
Cadmium	10
Lead	20
Cyanide	10
Alkalinity (CaCO <sub>3</sub> )	1000
Chloride	1000
Fluoride	200
Sulfate	5000
Ammonia Nitrogen	50
TKN	1000
Nitrate and Nitrite	50
TOC	5000
Total Phosphorus	10

-----  
 Other inorganic detection limits can be provided when requested.

TABLE 6

## METHOD DETECTION LIMITS FOR ORGANICS FROM CLP

VOLATILES	CAS NO.	DETECTION LIMITS*	
		LOW WATER <sup>a</sup> ug/L	LOW SOIL/SEDIMENT ug/Kg
Chloromethane	74-87-3	10	10
Bromomethane	74-83-9	10	10
Vinyl Chloride	75-01-4	10	10
Chloromethane	75-00-3	10	10
Methylene Chloride	75-09-2	5	5
Acetone	67-64-1	10	10
Carbon Disulfide	75-15-0	5	5
1,1-Dichloroethane	75-35-4	5	5
1,1-Dichloroethane	75-35-3	5	5
trans-1,2-Dichloroethene	156-60-5	5	5
Chloroform	67-66-3	5	5
1,2-Dichloroethane	107-06-2	5	5
2-Butanone	78-93-3	10	10
1,1,1-Trichloroethane	71-55-6	5	5
Carbon Tetrachloride	56-23-5	5	5
Vinyl Acetata	108-05-4	10	10
Bromodichloromethane	75-27-4	5	5
1,1,2,2-Tetrachloroethane	79-34-5	5	5
1,2-Dichloropropane	78-87-5	5	5
trans-1,3-Dichloropropene	10061-02-6	5	5
Trichloroethene	79-01-6	5	5
Dibromochloromethane	124-48-1	5	5
1,1,2-Trichloroethane	79-00-5	5	5
Benzene	71-43-2	5	5
cis-1,3-Dichloropropene	10061-01-5	5	5
2-Chloroethyl Vinyl Ether	100-75-8	10	10
Bromoform	75-25-2	5	5
2-Hexane	591-78-6	10	10
4-Methyl-2-pentanone	108-10-1	10	10
Tetrachloroethene	127-18-4	5	5
Toluene	108-88-3	5	5
Chlorobenzene	108-90-7	5	5
Ethyl Benzene	100-41-4	5	5
Styrene	100-42-5	5	5
Total Xylenes		5	5

<sup>a</sup> Medium Water Contract Required Detection Limits (CRDL) for Volatile HSL Compounds are 100 Times the Individual Low Water CRDL.

<sup>b</sup> Medium Soil/Sediment Contract Required Detection Limits (CRDL) for Volatile HSL Compounds are 100 Times the Individual Low Soil/Sediment CRDL.

Table 6 (Continued)

SEMI-VOLATILES	CAS NO.	DETECTION LIMITS*	
		LOW WATER <sup>c</sup> ug/L	LOW SOIL/SEDIMENT <sup>d</sup> ug/Kg
N-Nitrosodimethylamine	62-75-9	10	330
Phenol	108-95-2	10	330
Aniline	62-53-3	10	330
bis(2-Chloroethyl) ether	111-44-4	10	330
2-Chlorophenol	95-57-8	10	330
1,3-Dichlorobenzene	541-73-1	10	330
1,4-Dichlorobenzene	106-46-7	10	330
Benzyl Alcohol	100-51-6	10	330
1,2-Dichlorobenzene	95-50-1	10	330
2-Methylphenol	95-48-7	10	330
bis(2-Chloroisopropyl) ether	39638-32-9	10	330
4-Methylphenol	106-44-5	10	330
N-Nitroso-Dipropylamine	621-64-7	10	330
Hexachloroethane	67-72-1	10	330
Nitrobenzene	98-95-3	10	330
Isophorone	78-59-1	10	330
2-Nitrophenol	88-75-5	10	330
2,4-Dimethylphenol	105-67-9	10	330
Benzoic Acid	65-85-0	50	330
bis(2-Chloroethoxy) methane	111-91-1	10	330
2,4-Dichlorophenol	120-83-2	10	330
1,2,4-Trichlorobenzene	120-82-1	10	330
Naphthalene	91-20-3	10	330
4-Chloroaniline	106-47-8	10	330
Hexachlorobutadiene	87-68-3	10	330
4-Chloro-3-methylphenol (para-chloro-meta-cresol)	59-50-7	10	330
2-Methylnaphthalene	91-57-6	10	330
Hexachlorocyclopentadiene	77-47-4	10	330
2,4,6-Trichlorophenol	88-06-2	10	330
2,4,5-Trichlorophenol	95-95-4	50	1600
2-Chloronaphthalene	91-58-7	10	330
2-Nitroaniline	88-74-4	50	1600
Dimethyl Phthalate	131-11-3	10	330
Acenaphthylene	208-96-8	10	330
3-Nitroaniline	99-09-2	50	1600
Acenaphthene	83-32-9	10	330
2,4-Dinitrophenol	51-28-5	50	1600
4-Nitrophenol	100-02-7	50	1600
Dibenzofuran	132-64-9	10	330
2,4-Dinitrotoluene	121-14-2	10	330

<sup>c</sup> Medium Water Contract Required Detection Limits (CRDL) for Semi-Volatile HSL Compounds are 100 Times the Individual Low Water CRDL.

<sup>d</sup> Medium Soil/Sediment Contract Required Detection Limits (CRDL) for Semi-Volatile HSL Compounds are 60 Times the Individual Low Soil/Sediment CRDL.

Table 6 (Continued)

SEMI-VOLATILES	CAS NO.	DETECTION LIMITS*	
		LOW WATER <sup>c</sup> ug/L	LOW SOIL/SEDIMENT ug/Kg
2,6-Dinitrotoluene	606-20-2	10	330
Diethylphthalate	84-66-2	10	330
4-Chlorophenol Phenyl ether	7005-72-3	10	330
Fluorene	86-73-7	10	330
4-Nitroaniline	100-01-6	50	1600
4,6-Dinitro-2-methylphenol	534-52-1	50	1600
N-nitrosodiphenylamine	86-30-6	10	330
4-Bromophenyl Phenyl ether	101-55-3	10	330
Hexachlorobenzene	118-74-1	10	330
Pentachlorophenol	87-86-5	50	1600
Phenanthrene	85-01-8	10	300
Anthracene	120-12-7	10	330
Di-n-butylphthalate	84-74-2	10	330
Fluoranthene	206-44-0	10	330
Benzidine	92-87-5	100	1600
Pyrene	129-00-0	10	330
Butyl Benzyl Phthalate	85-68-7	10	330
3,3 -Dichlorobenzidine	91-94-1	20	660
Benzo(a)anthracene	56-55-3	10	330
bis(2-ethylehexyl)phthalate	117-81-7	10	330
Chrysene	218-01-9	10	330
Di-n-octyl Phthalate	117-84-0	10	330
Benzo(b)fluoranthene	205-99-2	10	330
Benzo(k)fluoranthene	207-08-9	10	330
Benzo(a)pyrene	50-32-8	10	330
Indeno(1,2,3-cd)pyrene	193-39-5	10	330
Dibenz(a,h)anthracene	53-70-3	10	330
Benzo(g,h,i)perylene	191-24-2	10	330

<sup>c</sup> Medium Water Contract Required Detection Limits (CRDL) for Semi-Volatile HSL Compounds are 100 Times the Individual Low Water CRDL.

<sup>d</sup> Medium Soil/Sediment Contract Required Detection Limits (CRDL) for Semi-Volatile HSL Compounds are 60 Times the Individual Low Soil/Sediment CRDL.

Table 6 (Continued)

PESTICIDES	CAS NO.	DETECTION LIMITS*	
		LOW WATER <sup>e</sup> ug/L	LOW SOIL/SEDIMENT <sup>f</sup> ug/Kg
alpha-BHC	319-84-6	0.05	2.0
beta-BHC	319-85-7	0.05	2.0
delta-BHC	319-86-8	0.05	2.0
gamma-BHC (Lindane)	58-89-9	0.05	2.0
Heptachlor	76-44-8	0.05	2.0
Aldrin	309-00-2	0.05	2.0
Heptachlor Epoxide	1024-57-3	0.05	2.0
Endosulfan I	959-98-8	0.05	2.0
Dieldrin	60-57-1	0.10	4.0
4,4 -DDE	72-55-9	0.10	4.0
Endrin	72-20-8	0.10	4.0
Endosulfan II	33213-65-9	0.10	4.0
4,4 -DDD	72-54-8	0.10	4.0
Endrin Aldehyde	7421-93-4	0.10	4.0
Endosulfan Sulfate	1031-07-8	0.10	4.0
4,4 -DDT	50-20-3	0.10	4.0
Endrin Ketone	53494-70-5	0.10	4.0
Methoxychlor	72-43-5	0.5	20.0
Chlordane	57-74-9	0.5	20.0
Toxaphene	8001-35-2	1.0	40.0
Aroclor 1016	12674-11-2	0.5	20.0
Aroclor 1221	11104-28-2	0.5	20.0
Aroclor 1232	11141-16-5	0.5	20.0
Aroclor 1242	53469-21-9	0.5	20.0
Aroclor 1248	12672-29-6	0.5	20.0
Aroclor 1254	11097-69-1	1.0	40.0
Aroclor 1260	11096-82-5	1.0	40.0

<sup>e</sup> Medium Water Contract Required Detection Limits (CRDL) for Pesticide HSL Compounds are 100 Times the Individual Low Water CRDL.

<sup>f</sup> Medium Soil/Sediment Contract Required Detection Limits (CRDL) for Pesticide HSL Compounds are 60 Times the Individual Low Soil/Sediment CRDL.

\*Detection limits listed for soil/sediment are based on net weight. The detection limits calculated by the laboratory for soil/sediment, calculated on dry weight basis, as required by the contract, will be higher.

Specific detection limits are highly matrix dependent. The detection limits listed herein are provided for guidance and may not always be achievable.

TABLE 7

## METHOD DETECTION LIMITS FOR INORGANICS FROM CLP

<u>ELEMENT</u>	<u>CONTRACT REQUIRED DETECTION LEVEL<sup>1,2</sup> (ug/L)</u>
Aluminum	200
Antimony	60
Arsenic	10
Barium	200
Beryllium	5
Cadmium	5
Calcium	5000
Chromium	10
Cobalt	50
Copper	25
Cyanide	10
Iron	100
Lead	5
Magnesium	5000
Manganese	15
Mercury	0.2
Nickel	40
Potassium	5000
Selenium	5
Silver	10
Sodium	5000
Thallium	10
Tin	40
Vanadium	50
Zinc	20

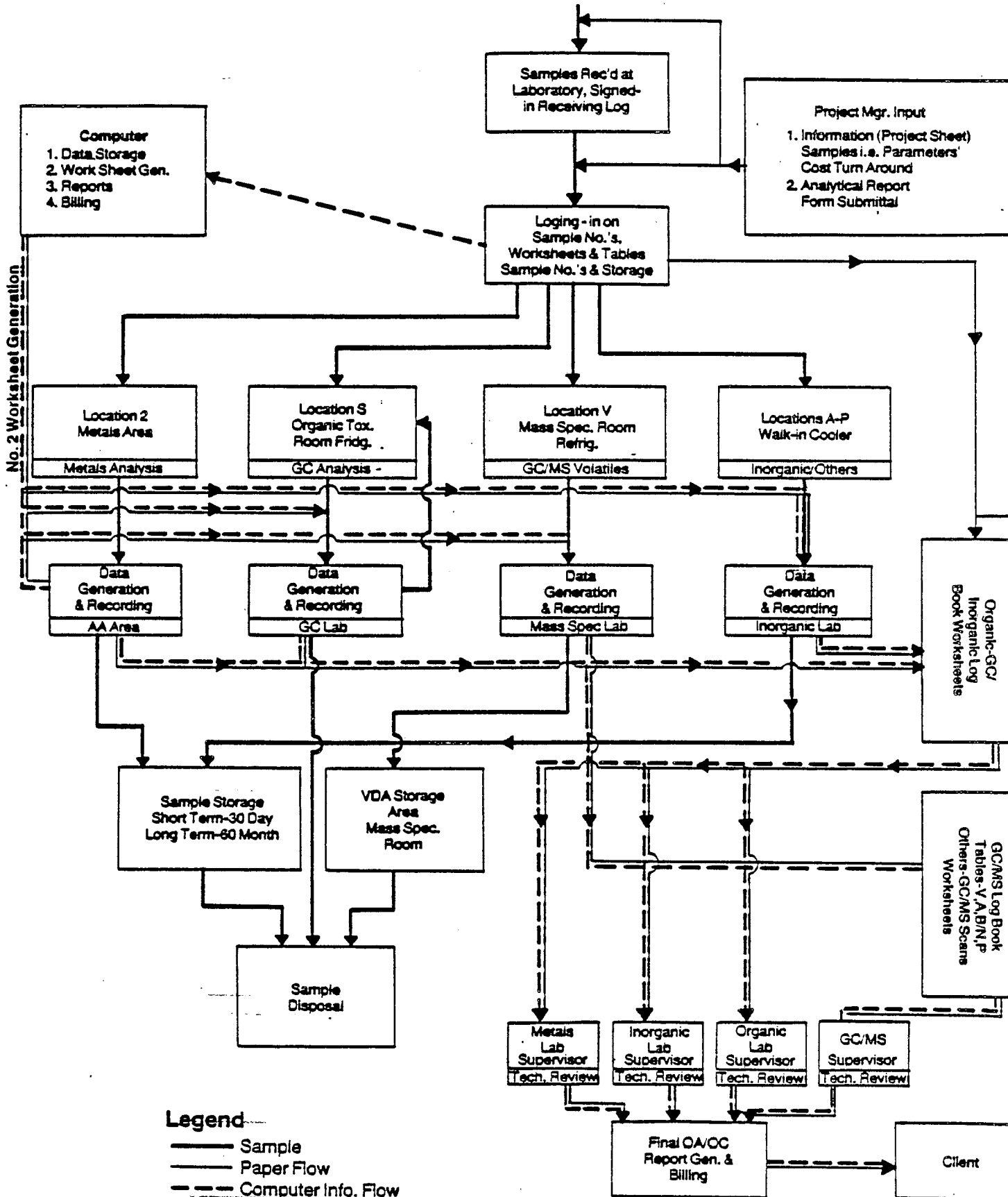
- 1: Any analytical method specified in SOW Exhibit D may be utilized as long as the documented instrument or method detection limits meet the Contract Required Detection Level (CRDL) requirements. Higher detection levels may only be used in the following circumstances:

If the sample concentration exceeds two times the detection limit of the instrument or method in use, the value may be reported even though the instrument or detection limit may not equal the contract required detection level.

- 2: These CRDL are the instrument detection limits obtained in pure water that must be met using the procedure in Exhibit E. The detection limits for samples may be considerably higher depending on the sample matrix.

# Sample & Document Flow Diagram

Figure 1







ROUTE TO: \_\_\_\_\_

DATE: \_\_\_\_\_  
TIME: \_\_\_\_\_

## EDI LABORATORY PROBLEM PROJECT REPORT

SAMPLES RECEIVED ON \_\_\_\_\_ AT \_\_\_\_\_ AM/PM FROM: \_\_\_\_\_  
AND DESCRIBED AS \_\_\_\_\_ WERE RECEIVED HAVING THE  
FOLLOWING DEFICIENCIES.

REQUEST FORM - ABSCENT/INCOMPLETE	<input type="checkbox"/>
CHAIN-OF-CUSTODY - ABSCENT/INCOMPLETE	<input type="checkbox"/>
CHAIN-OF-CUSTODY - DOES NOT MATCH SAMPLE TAGS	<input type="checkbox"/>
SAMPLE BOTTLES - BROKEN	<input type="checkbox"/>
SAMPLES ABSCENT - QUAN. DOES NOT MATCH REQUEST FORM	<input type="checkbox"/>
SAMPLE BOTTLES - INCORRECT FOR ANALYSIS	<input type="checkbox"/>
SAMPLE PRESERVATIVES - INCORRECT FOR ANALYSIS	<input type="checkbox"/>
SAMPLE VOLUMES - INCORRECT FOR ANALYSIS	<input type="checkbox"/>
SAMPLE TAGS - WRONG I.D./ABSCENT	<input type="checkbox"/>
FIELD FORMS - ABSCENT/INCOMPLETE	<input type="checkbox"/>
CUSTODY SEALS - ABSCENT/NOT INTACTED	<input type="checkbox"/>
SMO FORMS - ABSCENT	<input type="checkbox"/>
NON-ROUTINE PROJECT - NO PRIOR APPROVAL	<input type="checkbox"/>

THE SAMPLES IN QUESTION WILL BE \_\_\_\_\_ PROCESSED AS IS \_\_\_\_\_ PLACED ON HOLD \_\_\_\_\_  
UNTIL THE CORRECTIVE ACTIONS OR DIRECTIVES ARE ISSUED.

THANK YOU  
EDI LABORATORY  
SAMPLE COORDINATOR

-----















## STANDARD OPERATING PROCEDURE

### Sample Container, Cleaning Procedure Prior to Sampling:

TYPE	SIZE (ml)	ANALYSIS	CLEANING PROCEDURE
Plastic	125	Unspecified	D.I. Rinse
	250	Unspecified	D.I. Rinse
	500	Radiological	D.I. Rinse
	1000	Metals	HNO <sub>3</sub> , Rinse + D.I.
	1000	Cyanides	NaOH Rinse + D.I.
	1000	General Inorganics	D.I. Rinse
	1000	Nutrients	H <sub>2</sub> SO <sub>4</sub> Rinse + D.I.
Glass	125	Sulfide	D.I. Rinse
	125	TOC	D.I. Rinse + Bake*
	250	TOX	Solvent Rinse + Bake*
	500	Phenols	H <sub>2</sub> SO <sub>4</sub> Rinse + D.I.
	500	Organic	Solvent Rinse + Bake
	1000	Organic	Solvent Rinse + Bake
	40	VOA	D.I. + Bake or Purchase cleaned (Supelco)