

Table 5-47. Reporting Limit Data for Organonitrogen Pesticides, EPA 633

Parameter	Reporting Limits
	Aqueous* ( $\mu\text{g/L}$ )
Bromacil	2.5
Deet	1.0**
Hexazinone	2.5
Metribuzin	1.5
Terbacil	2.0**
Triadimefon	7.0**
Tricyclazole	1.0**

\*Based on the lowest standard that ESE routinely uses, taking into account the sample volume and final extract volume. The lowest standard is chosen to be within the range of 5 to 10 times the background noise of the instrument.

\*\*Ten times the detection limits listed in the Method EPA 633.

Source: ESE.

Table 5-48. Analytes, Precision, and Accuracy Data for Certain Amine Pesticides and Lethane, EPA 645

Parameter	Aqueous	
	Precision (RPD)	Accuracy (% Recovery)
Alachlor*	40	64-144
Butachlor	25	68-118
Diphenamid	43	57-143
Fluridone*	35	57-127
Lethane*	60	33-153
Norflurazon	22	68-112

Reference: Accuracy and Precision: EPA Method 645 -- Test Method for Organic Chemical Analysis of Municipal and Industrial Wastewater, EPA 600/4-82-057, July 1982.

\*Matrix spike and QC check sample compound.

Source: ESE.

Table 5-49. Reporting Limit Data for Certain Amine Pesticides and Lethane,  
EPA 645

Parameter	Reporting Limits Aqueous* (µg/L)
Alachlor	2.0
Butachlor	3.0
Diphenamid	2.0
Fluridone	5.0
Lethane	1.0
Norflurazon	0.2

\*Ten times the detection limits listed in the method, EPA 645.

Table 5-50. Analytes, Precision, and Accuracy Data for Nitroaromatics and Nitramines by High Performance Liquid Chromatography (HPLC). SW 8330

	Aqueous <sup>1</sup>		Solid <sup>1</sup>	
	Precision (RPD)	Accuracy (% Recovery)	Precision (RPD)	Accuracy (% Recovery)
HMX <sup>2</sup>	13	84-111	18	80-116
RDX <sup>2</sup>	30	51-111	18	71-107
1,3,5-Trinitrobenzene <sup>2</sup>	28	46-102	25	65-115
1,3-Dinitrobenzene <sup>2</sup>	37	58-132	30	70-130
Methyl-2,4,6-Trinitrophenylnitramine (Tetryl) <sup>2</sup>	21	67-109	46	65-157
Nitrobenzene <sup>2</sup>	32	44-108	24	72-120
2,4,6-Trinitrotoluene <sup>2</sup>	38	48-124	25	72-118
2,4-Dinitrotoluene <sup>2</sup>	21	60-102	19	68-106
2,6-Dinitrotoluene <sup>2</sup>	26	67-119	44	58-146
o-Nitrotoluene <sup>2</sup>	28	53-109	22	70-114
m-Nitrotoluene <sup>2</sup>	48	40-136	48	40-136
p-Nitrotoluene <sup>2</sup>	26	60-112	26	60-112

<sup>2</sup>Matrix spike and QC check sample compound

<sup>1</sup>Accuracy and precision criteria based on ESE historical data, unless specified differently.

<sup>2</sup>Accuracy and precision criteria based on ESE method validation studies.

Source: ESE.

Table 5-51. Reporting Limit Data for Nitroaromatics and Nitroamines by High Performance Liquid Chromatography, SW 8330

Parameter	Reporting Limit	
	Aqueous* ( $\mu\text{g/L}$ )	Solid† ( $\mu\text{g/kg}$ )
HMX	0.25	500
RDX	0.25	500
1,3,5-Trinitrobenzene	0.20	250
1,3-Dinitrobenzene	0.10	200
Methyl-2,4,6-Trinitro- phenylaitramine	0.20	300
Nitrobenzene	0.20	300
4-Amino-2,6-Dinitrotoluene	0.15	250
2,4,6-Trinitrotoluene	0.15	250
2-Amino-4,6-Dinitrotoluene	0.15	250
2,4-Dinitrotoluene	0.15	150
2,6-Dinitrotoluene	0.15	200
o-Nitrotoluene	0.25	500
m-Nitrotoluene	0.25	500
p-Nitrotoluene	0.30	600

\*Based on the lowest standard that ESE routinely uses, taking into account the sample volume and final extract volume. The lowest standard is chosen to be within the range of 50 to 10 times the background noise of the instrument.

†Based on the lowest standard that ESE routinely uses, taking into account the sample weight and final extract volume. The lowest standard is chosen to be within the range of 5 to 10 times the background noise of the instrument. The solid reporting limits are expressed on a wet weight basis.

Source: ESE.

Table 5-52. Analytes, Precision, and Accuracy Data for Nitroaromatics and Nitramines by High Performance Liquid Chromatography (HPLC), AEC QA Plan 11/92\*\*

	Aqueous <sup>1</sup>		Solid <sup>1</sup>	
	Precision (RPD)	Accuracy (% Recovery)	Precision (RPD)	Accuracy (% Recovery)
HMX <sup>††</sup>	13	84-111	18	80-116
RDX <sup>*</sup>	30	51-111	18	71-107
1,3,5-Trinitrobenzene <sup>*</sup>	28	46-102	25	65-115
1,3-Dinitrobenzene <sup>††</sup>	37	58-132	30	70-130
Methyl-2,4,6-Trinitrophenylnitramine (Tetryl) <sup>††</sup>	21	67-109	46	65-157
Nitrobenzene <sup>*</sup>	32	44-108	24	72-120
2,4,6-Trinitrotoluene <sup>*</sup>	38	48-124	23	72-118
2,4-Dinitrotoluene <sup>*</sup>	21	60-102	19	68-106
2,6-Dinitrotoluene	26	67-119	44	58-146
o-Nitrotoluene <sup>*</sup>	28	53-109	22	70-114
m-Nitrotoluene <sup>††</sup>	48	40-136	48	40-136
p-Nitrotoluene <sup>††</sup>	26	60-112	26	60-112

<sup>†</sup>Matrix spike and QC check sample compound

<sup>1</sup>Accuracy and precision criteria based on ESE historical data, unless specified differently.

<sup>††</sup>UW32 and LW12 are ESE's USATHAMA approved methods (see Appendices H and I).

<sup>\*</sup>Accuracy and precision criteria based on ESE method validation studies for Methods LW12 and UW32.

Source: ESE.

Table 5-53. Reporting Limit Data for Nitroaromatics and Nitroamines by High Performance Liquid Chromatography, AEC QA Plan 11/92

Parameter	Reporting Limit	
	Aqueous* (µg/L)	Solid† (µg/kg)
HMX	0.25	500
RDX	0.25	500
1,3,5-Trinitrobenzene	0.20	250
1,3-Dinitrobenzene	0.10	200
Methyl-2,4,6-Trinitro- phenylnitramine	0.20	300
Nitrobenzene	0.20	300
4-Amino-2,6-Dinitrotoluene	0.15	250
2,4,6-Trinitrotoluene	0.15	250
2-Amino-4,6-Dinitrotoluene	0.15	250
2,4-Dinitrotoluene	0.060	150
2,6-Dinitrotoluene	0.070	200
o-Nitrotoluene	0.25	500
m-Nitrotoluene	0.25	500
p-Nitrotoluene	0.30	600

\*Based on the lowest standard that ESE routinely uses, taking into account the sample volume and final extract volume. The lowest standard is chosen to be within the range of 50 to 10 times the background noise of the instrument.

†Based on the lowest standard that ESE routinely uses, taking into account the sample weight and final extract volume. The lowest standard is chosen to be within the range of 5 to 10 times the background noise of the instrument. The solid reporting limits are expressed on a wet weight basis.

Source: ESE.

Table 5-54. Analyte, Precision, and Accuracy Data for Glyphosate, EPA 547 (Modified)

Parameter	Aqueous	
	Precision (RPD)	Accuracy (% Recovery)
Glyphosate	15	90-126

-Precision and accuracy are based on method validation study performed by ESE (see Appendix V).

Source: ESE.

Table 5-55. Reporting Limit Data for Glyphosate. EPA 547 (Modified)

Parameter	Reporting Limit Aqueous* (µg/L)
Glyphosate	2.5

\*Based on the lowest standard that ESE routinely uses, taking into account the sample volume and final extract volume. The lowest standard is chosen to be within the range of 5 to 10 times the background noise of the instrument.

Source: ESE.

Table 5-36. Analyte, Precision, and Accuracy Data for Ethylene-Bis-Dithiocarbamates (EBDC). EPA 630.1 (Modified)

	Aqueous*		Solid*	
	Precision (RPD)	Accuracy (% Recovery)	Precision (RPD)	Accuracy (% Recovery)
EBDC	9	92-100	12	67-91

\*Precision and accuracy are based on method validation studies performed by ESE (see Appendix W).

Source: ESE.

Table 5-57: Reporting Limit Data for Ethylene-Bis-Dithiocarbamates (EBDC), EPA 630.1 (Modified)

Parameter	Reporting Limit	
	Aqueous* ( $\mu\text{g/L}$ )	Solid† ( $\mu\text{g/kg}$ )
EBDC	5.5	100

\*Based on the lowest standard that ESE routinely uses, taking into account the sample volume and final extract volume. The lowest standard is chosen to be within the range of 5 to 10 times the background noise of the instrument.

Source: ESE.

Table 5-58. Analytes, Precision, and Accuracy Data for Organochlorine Pesticides and PCBs in Water, EPA 617

Parameter	Aqueous	
	Precision (RPD)	Accuracy (% Recovery)
Aldrin <sup>a</sup>	45 <sup>a</sup>	37 - 127 <sup>b</sup>
BHC, A	30	37 - 134
BHC, B	30	17 - 147
BHC, D	27	68 - 122
BHC, G (lindane) <sup>a</sup>	51 <sup>a</sup>	45 - 145 <sup>a</sup>
Chlordane	30	45 - 119
DDD, PP <sup>a</sup>	15	79 - 109
DDE, PP <sup>a</sup>	11	79 - 101
DDT, PP <sup>a</sup>	14	77 - 105
Dieldrin	15	85 - 113
Endosulfan, A	23	80 - 124
Endosulfan, B	14	79 - 107
Endosulfan sulfate	30	26 - 144
Endrin <sup>a</sup>	60 <sup>a</sup>	35 - 155 <sup>a</sup>
Endrin aldehyde	40 <sup>a</sup>	58 - 138 <sup>a</sup>
Heptachlor <sup>a</sup>	33 <sup>a</sup>	48 - 124 <sup>a</sup>
Heptachlor epoxide	12	82 - 106
Methoxychlor	20	77 - 117
Toxaphene	30	41 - 126
Mirex	14	75 - 103
Trifluralin	32	62 - 126
PCNB	19	64 - 102
PCB-1061 <sup>d</sup>	30	50 - 114
PCB-1221	30	15 - 178
PCB-1232	30	10 - 215
PCB-1242	30	39 - 150
PCB-1248	30	38 - 158
PCB-1254	30	29 - 131
PCB-1260 <sup>d</sup>	30	8 - 127
Dibutylchlorodate <sup>a</sup>	NA	46 - 146 <sup>b</sup>

Table S-58. Analytes, Precision, and Accuracy Data for Organochlorine Pesticides and PCBs in Water, EPA 617 (Continued, Page 2 of 2)

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Reference: Accuracy and Precision: EPA Methods 617 and SW 8080.

Note: NA = not applicable.

\*Matrix spike and QC check sample compound.

<sup>b</sup>Accuracy and precision criteria are based on ESE historical data.

<sup>c</sup>Surrogate; the surrogate is added to all environmental samples and quality control samples.

<sup>d</sup>PCB 1016 and PCB 1260 are only used as matrix spike and QCC samples compounds when using EPA 608/8080 to evaluate PCBs only.

Source: ESE.

Table 5-59. Reporting Limit Data for Organochlorine Pesticides and PCBs in Water,  
EPA 617

Parameter	Reporting Limits
	Aqueous* ( $\mu\text{g/L}$ )
Aldrin	0.006
BHC,A	0.006
BHC,B	0.006
BHC,D	0.006
BHC,G(lindane)	0.006
Chlordane	0.030
DDD,PP	0.006
DDE,PP	0.006
DDT,PP	0.006
Dieldrin	0.006
Endosulfan,A	0.006
Endosulfan,B	0.006
Endosulfan sulfate	0.006
Endrin	0.006
Endrin aldehyde	0.006
Heptachlor	0.006
Heptachlor epoxide	0.006
Methoxychlor	0.006
Toxaphene	0.6
PCB-1016	0.12
Mirex	0.2
Trifluralin	0.1
PCNB	0.02

Table 5-59. Reporting Limit Data for Organochlorine Pesticides and PCBs in Water, EPA 617 (Continued, Page 2 of 2)

Parameter	Reporting Limits Aqueous* ( $\mu\text{g/L}$ )
PCB-1221	0.12
PCB-1232	0.12
PCB-1242	0.12
PCB-1248	0.12
PCB-1254	0.12
PCB-1260	0.12

\*Based on the lowest standard that ESE routinely uses, taking into account the sample volume and final extract volume. The lowest standard is chosen to be within the range of 5 to 10 times the background noise of the instrument.

Source: ESE.

Table 5-60. Analyte, Precision, and Accuracy Data for Tetrazene, EPA 8331

Parameter	Aqueous*		Solid*	
	Precision (RPD)	Accuracy (% Recovery)	Precision (RPD)	Accuracy (% Recovery)
Tetrazene	10	94-114	25	35-85

\*Precision and accuracy are based on EPA Method 8331--Test Methods for Evaluating Solid Waste, EPA SW-846, 3rd Edition, September 1986.

Source: ESE.

Table 5-61. Reporting Limit Data for Tetrazene, EPA 8331

Parameter	Reporting Limit	
	Aqueous* ( $\mu\text{g/L}$ )	Solid ( $\mu\text{g/kg}$ )
Tetrazene	7	1,000

Source: ESE.

## 6.0 SAMPLE HANDLING PROCEDURES

### 6.1 INTRODUCTION

The validity of analytical data is dependent on the integrity of the field procedures employed in obtaining a sample. Environmental sampling has many variables which can affect analytical results. The properties of most contaminated materials warrant the analysis of a small aliquot of the bulk of the material. Proper sampling techniques must be employed to obtain a representative sample of the bulk material. For a sample to be representative, it must be collected and handled so as to keep its original physical form and chemical composition as much as possible and to protect against any loss or contamination. To achieve this sample's integrity, quality assurance procedures should be closely followed throughout the sampling effort.

Exhaustive and sometimes expensive actions are taken based on the analytical data generated from field sampling programs. Therefore, it is in the best interest of the investigation, as well as the public, to ensure the quality of the data by ensuring the quality of the samples being delivered to the analyst.

This section of the LCQAP details sample handling requirements in the laboratory.

### 6.2 SAMPLE CONTAINERS CLEANING PROCEDURES

#### 6.2.1 CLEANING PROCEDURES

ESE uses commercially cleaned sample containers whenever practical. At a minimum, only Type 200 series precleaned sample containers will be used. For HAZWRAP and other projects, as required, only sample containers provided with certificate of cleanliness will be used and the certificates will be kept on file. Table 6-1 summarizes the application of these cleaning procedures. If containers are cleaned and prepared inhouse, the same procedures will be followed. Cleaned sample containers are stored in a secured

Table 6-1. Sample Container Cleaning Procedures Within the Laboratory

Analysis/Parameter	Container Type	Matrix	Fraction Code	Cleaning Protocol*
Organic extractables include GC, HPLC, GC/MS, and Total Phenols Analyses	Glass jar with Teflon <sup>®</sup> -lined cap	Water	MS, EC, HB, UP, NP, LC, W, Z	A
	Glass jar with Teflon <sup>®</sup> -lined cap	Soil/Sediment	SS	A
	Aluminum foil and plastic bags	Tissue*	TS	NA
Organic purgeables including GC and GC/MS Analyses, TON, Aldicarb	Glass septum vial with Teflon <sup>®</sup> -lined cap	Water	V, VP, ED, AL, NP	B
	Wide-mouth glass jar with Teflon <sup>®</sup> -lined cap	Soil	SV	B
	Aluminum foil and plastic bags	Tissue*	TS	NA
Metals	Linear polyethylene container with polyethylene cap	Water	N	C
	Glass jar with Teflon <sup>®</sup> -lined cap (or new plastic)	Soil/Sediment	SS	A
	Plastic bags	Tissue*	TS	NA
Inorganics include total cyanide, alkalinity, acidity, residues, BOD, color, MBAS, COD, TOC, chloride, turbidity, sulfate, bromide, sulfide, fluoride, nutrients, and radionuclides	Linear polyethylene container with polyethylene cap	Water	C, B, S, H, R	D*
	Glass jar with Teflon <sup>®</sup> -lined cap (or new plastic)	Soil	SS	A
	Aluminum foil and plastic bags	Tissue*	TS	NA
Oil and grease (O&G), odor	Glass jar with Teflon <sup>®</sup> -lined cap	Water	O, OD	A
Oil and grease (O&G)	Glass jar with Teflon <sup>®</sup> -lined cap	Soil/Sediment	SS	A

Table 6-1. Sample Container Cleaning Procedures Within the Laboratory (Continued, Page 2 of 2)

Note:	Cleaning Protocol				Soiled/contaminated
	A	B	C	D	
BOD = biochemical oxygen demand. COD = chemical oxygen demand. GC/MS = gas chromatography/mass spectrometry. GC/HPLC = gas chromatography/high performance liquid chromatography. Glass = amber for all organic water analyses. MBAS = methylene blue active substance. NA = not applicable. TOC = total organic carbon. TOX = total organic halides.					
*Tissue samples for these parameters are first wrapped in aluminum foil and then put in plastic bags.					
	X	X	X		Wash with hot tap water using laboratory-grade, interference free, nonphosphate detergent.
	X	X	X		Rinse 3 times with tap water.
	X		X		Rinse with 1:1 nitric acid reagent-grade nitric acid diluted with ASTM Type 1 deionized water.
	X	X	X		Rinse 3 times with ASTM Type 1 deionized water.
	N				Rinse with pesticide-grade methylene chloride using 20 mL per 64-oz bottle, 10 mL per 32- or 16-oz bottle, or 5 mL per 8- or 4-oz bottle. Methylene chloride is used as organics fuser.
	X	X			Oven dry, using a forced-air oven at 105 to 125 C for 1 hour.
				X	Invert and air-dry in a contaminant-free environment.
	N	N	N		The containers are sealed with caps containing Teflon® liners or Teflon®-backed septa that had been cleaned the same way as the containers, packed in cartons, and stored until needed.
				X	No cleaning required; use new subainers (only).

Note: Cleaning protocols A, B, and C are applied by commercial supplier.  
 Cleaning protocol D is applied by ESE.

Source: ESE.

storage building away from the analytical laboratory until needed. Occasional audits of containers to document freedom from contaminants will be performed to supplement the various blanks that are frequently and routinely analyzed to provide similar QC data. Activities and records associated with contaminant-free containers are maintained.

All sample containers, either commercially cleaned or prepared inhouse, are stored in a secured storage building located away from the laboratory. When containers are needed, they are moved to the sample kit preparation area that is also located away from the laboratory and packed for shipment to the project site. Upon receipt of precleaned sample containers, the purchase order form is dated with date of receipt by the laboratory purchasing personnel and the purchase order form is filed. Documentation associated with the sample containers such as lot numbers and certification statements for 300 series containers are maintained and filed in their department by the kit preparation personnel. These containers are labeled individually with lot numbers, hence, it is not necessary to maintain records of lot numbers used for a particular project.

#### 6.2.2 TYPES OF WATER

DI water is defined as ESE water that has been treated by passing it through a standard resin column and an activated carbon unit. The water contains no detectable (i.e., ESE's routine detection limits) heavy metals or inorganic compounds of analytical interest and is relatively free of organic compounds. The water is acceptable for use in the initial rinsing of laboratory glassware and field equipment. Ultrapure water, used for equipment and field blanks, is defined as ESE DI water that has been additionally treated through a Milli-Q<sup>3</sup> treatment system and contains no organic compounds of analytical interest above ESE's routine detection limits. Organic-free water, used for trip blanks, is prepared by purging American Society for Testing and Materials (ASTM) Type 2 water at 60°C for 24 hours with Grade 6 helium.

Documentation is maintained to demonstrate reliability and "purity" of analyte free water source(s).

DI water other than ESE-treated water may be used if it is of documented equivalent quality. Use of commercially DI or distilled water is discouraged because it often contains phthalate esters.

### 6.3 SAMPLING CONTAINERS, VOLUMES, HOLDING TIMES AND PRESERVATION

#### 6.3.1 CONTAINERS AND SAMPLE HOLDING TIMES

Table 6-2 identifies the proper containers, preservation techniques, and maximum holding times established by EPA (40 CFR Part 136). The maximum holding times in Table 6-1 apply to water and soils as noted. Samples that exceed the regulatory holding times will be flagged by the laboratory coordinator in the final deliverable.

#### 6.3.2 SAMPLE PRESERVATION

Sample preservation is generally performed in the field. However, sample containers for volatile analysis (water only) are sent to the field with preservatives added to the containers. Sample preservation requirements are listed in Table 6-2.

Grades of the preservatives used are specified as a footnote in Table 6-2. Fresh preservatives are obtained from laboratory stocks prior to each sampling event.

### 6.4 SAMPLE SHIPPING FROM THE FIELD TO THE LABORATORY

A typical environmental sample consists of some type of soil or water matrix; however, other types of samples such as tissues or dust wipes are also collected. Whatever the field sample type, the field crew should package each sample container to ensure its integrity inside the shipping container. This packaging may include packing materials such as Bubble Wrap<sup>®</sup> or styrofoam fillers.

Table 6-2. Required Containers, Preservation Techniques, and Holding Times

Measurement	Container <sup>1</sup>	Preservation	Maximum Holding Time <sup>2</sup> (Waters and Soils)
<u>Metals</u>			
Chromium VI	P, G	Cool, 4°C	24 hours <sup>3</sup>
Mercury	P, G	HNO <sub>3</sub> to pH < 2	28 days
Metals, except chromium VI and mercury <sup>4</sup> (filtered and unfiltered)	P, G	HNO <sub>3</sub> to pH < 2	6 months
<u>Inorganic Tests</u>			
Acidity	P, G	Cool, 4°C	14 days <sup>3</sup>
Alkalinity	P, G	Cool, 4°C	14 days <sup>3</sup>
Ammonia	P, G	Cool, 4°C, H <sub>2</sub> SO <sub>4</sub> to pH < 2	28 days <sup>3</sup>
BOD	P, G	Cool, 4°C	48 hours <sup>3</sup>
Bromide	P, G	None required	28 days <sup>3</sup>
BOD, carbonaceous	P, G	Cool, 4°C	48 hours <sup>3</sup>
COD	P, G	Cool, 4°C, H <sub>2</sub> SO <sub>4</sub> to pH < 2	28 days <sup>3</sup>
Chloride	P, G	None required	28 days <sup>3</sup>
Chlorine, total	P, G	None required	Analyze immediately <sup>3,5</sup>
Color	P, G	Cool, 4°C	48 hours <sup>3</sup>
Cyanide, total and amenable to chlorination	P, G	Cool, 4°C, NaOH to pH > 12, 0.6 g ascorbic acid <sup>6</sup>	14 days <sup>3</sup>
Fluoride	P	None required	28 days <sup>3</sup>
Hardness	P, G	HNO <sub>3</sub> to pH < 2, H <sub>2</sub> SO <sub>4</sub> to pH < 2	6 months <sup>3</sup>
Hydrogen ion (pH)	P, G	Cool, 4°C	Analyze immediately <sup>3</sup>
Kjeldahl and organic nitrogen	P, G	Cool, 4°C, H <sub>2</sub> SO <sub>4</sub> to pH < 2	28 days <sup>3</sup>
Nitrate	P, G	Cool, 4°C	48 hours <sup>3</sup>
Nitrate (drinking water)			
Chlorinated	P, G	Cool, 4°C	28 days
Unchlorinated <sup>7</sup>	P, G	H <sub>2</sub> SO <sub>4</sub> to pH < 2	14 days
Nitrate-nitrite	P, G	Cool, 4°C, H <sub>2</sub> SO <sub>4</sub> to pH < 2	28 days <sup>3</sup>
Nitrite	P, G	Cool, 4°C	48 hours <sup>3</sup>
Oil and grease	G	Cool, 4°C, H <sub>2</sub> SO <sub>4</sub> to pH < 2	28 days <sup>3</sup>
Organic carbon	P, G	Cool, 4°C, HCl or H <sub>2</sub> SO <sub>4</sub> to pH < 2	28 days <sup>3</sup>

Table 6-2. Required Containers, Preservation Techniques, and Holding Times (Continued, Page 2 of 6)

Measurement	Container <sup>1</sup>	Preservation	Maximum Holding Time <sup>2</sup> (Waters and Soils)
<u>Inorganics (cont)</u>			
Orthophosphate	P, G	Filter immediately, cool, 4°C	48 hours <sup>3</sup>
Oxygen, dissolved (DO)	G Bottle and top	None required	Analyze immediately <sup>3</sup>
Probe	G Bottle and top	Fix onsite and store in dark	8 hours <sup>3</sup> Phenols
Winkler	G only	Cool, 4°C, H <sub>2</sub> SO <sub>4</sub> to pH < 2	28 days <sup>3</sup>
Phosphorus (elemental)	G	Cool, 4°C	48 hours <sup>3</sup>
Phosphorus, total	P, G	Cool, 4°C, H <sub>2</sub> SO <sub>4</sub> to pH < 2	28 days <sup>3</sup>
MBAS	P, G	Cool, 4°C	48 hours <sup>3</sup>
Bromates (ion chromatography)	P, G	Cool, 4°C	30 days <sup>3</sup>
Corrosivity (calculated)	P, G	Cool, 4°C	7 days <sup>3</sup>
Odor	G	Cool, 4°C	6 hours <sup>3</sup>
Unionized Ammonia (calculated)	P, G	Cool, 4°C Na <sub>2</sub> SO <sub>4</sub>	8 hours <sup>3</sup> 28 days <sup>3</sup>
Residue, total	P, G	Cool, 4°C	7 days <sup>3</sup>
Residue, filterable	P, G	Cool, 4°C	7 days <sup>3</sup>
Residue, nonfilterable (TSS)	P, G	Cool, 4°C	7 days <sup>3</sup>
Residue, settleable	P, G	Cool, 4°C	48 hours <sup>3</sup>
Residue, volatile	P, G	Cool, 4°C	7 days <sup>3</sup>
Silica	P	Cool, 4°C	28 days <sup>3</sup>
Specific conductance	P, G	Cool, 4°C	28 days <sup>3</sup>
Sulfate	P, G	Cool, 4°C	28 days <sup>3</sup>
Sulfide	P, G	Cool, 4°C, add zinc acetate plus NaOH to pH > 9	7 days <sup>3</sup>
Sulfite	P, G	None required	Analyze immediately <sup>3</sup>
Surfactants	P, G	Cool, 4°C	48 hours <sup>3</sup>
Temperature	P, G	None required	Analyze immediately <sup>3</sup>
Turbidity	P, G	Cool, 4°C	48 hours <sup>3</sup>
<u>Organic Tests</u>			
Purgeable halocarbons	G, Teflon <sup>®</sup> -lined septum	Cool, 4°C, 0.008N Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> <sup>3</sup> store in dark	14 days

Table 6-2. Required Containers, Preservation Techniques, and Holding Times (Continued, Page 3 of 6)

Measurement	Container <sup>1</sup>	Preservation	Maximum Holding Time <sup>2</sup> (Waters and Soils)
Organics (cont)			
Perceptible aromatic hydrocarbons	G. Teflon <sup>®</sup> -lined septum	Cool, 4°C. DUMKIS <sup>®</sup> Na <sub>2</sub> S <sub>2</sub> O <sub>5</sub> <sup>1</sup> HCl to pH 2	14 days
Arochlor and acrylonitrile	G. Teflon <sup>®</sup> -lined septum	Cool, 4°C. DUMKIS <sup>®</sup> Na <sub>2</sub> S <sub>2</sub> O <sub>5</sub> <sup>1</sup> Adjust pH to 4-5	14 days
Phenols	G. Teflon <sup>®</sup> lined cap	Cool, 4°C. DUMKIS <sup>®</sup> Na <sub>2</sub> S <sub>2</sub> O <sub>5</sub> <sup>1</sup> store in dark	7/200 days for waters <sup>1</sup> 14/200 days for soils <sup>1</sup> 7/400 days for waters <sup>1</sup>
Benzidines	G. Teflon <sup>®</sup> -lined cap	Cool, 4°C. DUMKIS <sup>®</sup> Na <sub>2</sub> S <sub>2</sub> O <sub>5</sub> <sup>1</sup> store in dark	7/400 days for waters <sup>1</sup> 14/200 days for soils <sup>1</sup>
Phthalate esters	G. Teflon <sup>®</sup> -lined cap	Cool, 4°C. store in dark	7/400 days for waters <sup>1</sup> 14/200 days for soils <sup>1</sup>
Nitrosamines	G. Teflon <sup>®</sup> -lined cap	Cool, 4°C. DUMKIS <sup>®</sup> Na <sub>2</sub> S <sub>2</sub> O <sub>5</sub> <sup>1</sup> store in dark	7/400 days for waters <sup>1</sup> 14/200 days for soils <sup>1</sup>
PCBs, pesticides, herbicides	G. Teflon <sup>®</sup> -lined cap	Cool, 4°C. DUMKIS <sup>®</sup> Na <sub>2</sub> S <sub>2</sub> O <sub>5</sub> <sup>1</sup> store in dark	7/400 days for waters <sup>1</sup> 14/200 days for soils <sup>1</sup>
Nitroaromatics and isophenols	G. Teflon <sup>®</sup> -lined cap	Cool, 4°C. DUMKIS <sup>®</sup> Na <sub>2</sub> S <sub>2</sub> O <sub>5</sub> <sup>1</sup> store in dark	7/400 days for waters <sup>1</sup> 14/200 days for soils <sup>1</sup>
Polynuclear aromatic hydrocarbons	G. Teflon <sup>®</sup> -lined cap	Cool, 4°C. DUMKIS <sup>®</sup> Na <sub>2</sub> S <sub>2</sub> O <sub>5</sub> <sup>1</sup> store in dark	7/400 days for waters <sup>1</sup> 14/200 days for soils <sup>1</sup>
Halobethers	G. Teflon <sup>®</sup> -lined cap	Cool, 4°C. DUMKIS <sup>®</sup> Na <sub>2</sub> S <sub>2</sub> O <sub>5</sub> <sup>1</sup> store in dark	7/400 days for waters <sup>1</sup> 14/200 days for soils <sup>1</sup>
Volatile organics	G. Teflon <sup>®</sup> -lined septum	Cool, 4°C. DUMKIS <sup>®</sup> Na <sub>2</sub> S <sub>2</sub> O <sub>5</sub> <sup>1</sup> HCl to pH 2	14/200 days for waters <sup>1</sup> 14 days
EDB, DBCP	G. Teflon <sup>®</sup> -lined septum	Cool, 4°C. DUMKIS <sup>®</sup> Na <sub>2</sub> S <sub>2</sub> O <sub>5</sub> <sup>1</sup>	28 days
Chlorinated hydrocarbons	G. Teflon <sup>®</sup> -lined cap	Cool, 4°C. store in dark	7/400 days for waters <sup>1</sup> 14/200 days for soils <sup>1</sup>
TCDD	G. Teflon <sup>®</sup> -lined cap	Cool, 4°C. DUMKIS <sup>®</sup> Na <sub>2</sub> S <sub>2</sub> O <sub>5</sub> <sup>1</sup> store in dark	7/400 days for waters <sup>1</sup> 14/200 days for soils <sup>1</sup>
Total organic halogens (TOX)	G. Teflon <sup>®</sup> -lined cap	Cool, 4°C. H <sub>2</sub> SO <sub>4</sub> to pH < 2 store in dark	14/200 days for waters <sup>1</sup> 28 days <sup>1</sup>

Table 6-2. Required Containers, Preservation Techniques, and Holding Times (Continued, Page 4 of 6)

Measurement	Container <sup>1</sup>	Preservation	Maximum Holding Time <sup>2</sup> (Waters and Soils)
<u>Organics (cont)</u>			
Acid and base/neutral extractables	G, Teflon <sup>®</sup> -lined cap	Cool, 4°C, 0.008% Na <sub>2</sub> S <sub>2</sub> O <sub>5</sub> <sup>3</sup> store in dark	7/40 days for waters <sup>4</sup> 14/40 days for soils <sup>4</sup>
Nitroaromatics and Nitramines	G, Teflon <sup>®</sup> -lined cap	Cool, 4°C	7/20 days for waters <sup>4</sup> 14/40 days for soils <sup>4</sup>
<u>Radiological Tests</u>			
Alpha, beta, Sr-90, Ra-226, Ra-228, Uranium, photon emitters	P, G	HCL, HNO <sub>3</sub> , to pH < 2	6 months
Cesium-134, Iodine-131, Titanium	P, G	None	6 months
<u>Tissues</u>			
Organics, inorganics and radiological tests	Aluminum foil and plastic bag	Freeze, -20° C or below	12 months
Metals tests	Plastic bag	Freeze, -20° C or below	12 months

Note: BOD = biochemical oxygen demand,  
 COD = chemical oxygen demand,  
 G = glass,  
 HCl = hydrochloric acid (metals grade),  
 HNO<sub>3</sub> = nitric acid (metals grade),  
 H<sub>2</sub>SO<sub>4</sub> = sulfuric acid (metals grade),  
 NS = none specified by EPA.

Na<sub>2</sub>SO<sub>5</sub> = sodium sulfite (ACS grade),  
 Na<sub>2</sub>S<sub>2</sub>O<sub>5</sub> = sodium thiosulfate (ACS grade),  
 P = polyethylene,  
 PCB = polychlorinated biphenyl,  
 NaOH = sodium hydroxide (ACS grade),  
 °C = degrees Celsius.

Table 6-2. Required Containers, Preservation Techniques, and Holding Times (Continued, Page 5 of 6)

- <sup>1</sup> For nonvolatile organics, containers for soil and sediment samples are glass with Teflon<sup>®</sup>-lined caps and for volatiles, containers are glass with Teflon<sup>®</sup>-lined septum.
- <sup>2</sup> Soil sample containers for inorganics are glass jars with Teflon<sup>®</sup>-lined caps, polyethylene (P), or glass (G).
- <sup>3</sup> Samples should be analyzed as soon as possible after collection. The times listed are the maximum times that samples may be held before analysis and still considered valid. Samples may be held for longer periods only if the laboratory has data on file to show that the specific types of samples under study are stable for the longer time.
- <sup>4</sup> Holding times provided are for waters. EPA does not have holding times for these parameters in soil. These water holding times will be used as goals for those methods where a soil analysis is applicable.
- <sup>5</sup> 7/20 = 7 days until extraction; 20 days from extraction until analysis. 14/20 = 14 days until extraction; 20 days from extraction until analysis.
- <sup>6</sup> Sample preservation should be performed immediately upon sample collection. The only preservation for soil samples is cooling at 4°C. For composite samples, each aliquot should be preserved at the time of collection. When use of an automatic sampler makes it impossible to preserve each aliquot, samples may be preserved by maintaining at 4°C until compositing and sample splitting are completed (maximum allowable time is 20 hours). Na<sub>2</sub>S<sub>2</sub>O<sub>4</sub> is used only in the presence of residual chlorine.
- <sup>7</sup> If residual chlorine is present, sodium thiosulfate is added to the sample vial first, the vial is then filled to almost full volume with sample, acid is added, and finally the vial is filled. Note: It is not recommended to mix the two preservatives (and sample) together in an intermediate vessel.
- <sup>8</sup> These parameters are best analyzed in the field. In consideration of shipping limitations, when these analyses are requested of our laboratory for confirmation purposes, ESE's policy is to analyze these constituents within 24 hours of receipt.
- <sup>9</sup> The following test should be performed for cyanide samples:
  - (a) Oxidizing agents--Test the sample using KI-starch paper. If present, add a few crystals of ascorbic acid and test until negative. Add an additional 0.6 gram of ascorbic acid for each liter of sample to remove the chlorine.
  - (b) Sulfides--When sulfide is present as indicated by a positive test with lead acetate paper, the maximum holding time is 24 hours. Remove the sulfides by (1) filtration of sample if visible particulates are present, (2) precipitation with cadmium nitrate until a negative spot test is obtained, (3) filtration of the precipitate, and (4) addition of NaOH to pH > 12 if sulfides are not removed with the previous procedure.
- <sup>10</sup> Temperature and pH must be measured onsite at the time of sample collection. Seven days is the maximum time for laboratory analysis of total alkalinity, calcium ion, and total solids.
- <sup>11</sup> The results of the measurements of pH, temperature, salinity (if applicable), and the ammonium ion concentration in the sample are used to calculate the concentration of ammonia in the unionized state. Temperature, pH, and salinity must be measured onsite at the time of sample collection. Laboratory analysis of the ammonium ion concentration should be conducted within 8 hours of sample collection. If prompt analysis of ammonia is impossible, preserve samples with H<sub>2</sub>SO<sub>4</sub> to pH between 1.5 and 2. Acid-preserved samples, stored at 4°C, may be held up to 28 days for ammonia determination. Sodium thiosulfate should only be used if fresh samples contain residual chlorine.
- <sup>12</sup> Chlorinated means that the source water has a detectable amount of residual chlorine, as will be indicated by the field test.
- <sup>13</sup> Nonchlorinated means that the source water contains no detectable amount of residual chlorine (i.e., below the detection limit).

Source: ESE.



Sample containers are typically shipped by bonded courier to the laboratory. Samples are shipped by overnight delivery as soon as possible after collection (usually daily), with receiving signature required. Sample receipt and check-in at the laboratory is performed by the sample custodian, as described in Section 7.3.

Samples are usually organized by sample location in each shipping container with all of the fractions collected from a given station grouped together. A possible exception to this procedure would include the collection of large quantities of samples for VOC analyses.

If the samples require chilling/freezing, the sample containers will be isolated from the chilling/freezing materials using appropriate, waterproof materials such as plastic garbage bags. Typically, wet ice is used to chill the samples; reusable blue ice-type chilling products will not be used due to possible chemical interferences. If a sample must be kept frozen in a solid state, dry ice is used.

The chain-of-custody logsheet for the samples in each shipping container is sealed in a plastic Ziploc<sup>®</sup> bag and taped to the inside of the container. ESE's policy requires sealing all sample shipping containers with evidence tape prior to shipping.

#### 6.5 REAGENT AND STANDARD STORAGE

Storage requirements of reagents and standards used are presented in Table 6-3.

Table 6-3. Reagent Storage

Reagent	Method of Storage
Solvents	Stored in original containers in a vented storage room, or stored in double-walled flammable liquid storage cabinets. Stockroom personnel check the storage cabinets daily and transfer solvents from the storage room to the storage cabinets as needed. Note: Methanol used for volatile organic analyses are stored in the GC-Volatiles and GC/MS-Volatiles analysis areas.
Inorganic acids	Stored in original containers in the ESE stockroom. Once issued from the stockroom to a department, the acids are kept in safety carriers and stored along with the carriers in the department's cabinet designated for acids only.
Organic acids	Stored in original containers in the ESE stockroom. Once issued from the stockroom to a department, the acids are kept in safety carriers and stored along with the carriers in the department's cabinet designated for acids only. Note: Organic acids are stored in separate cabinets from the inorganic acids.
Causities	Stored in original containers in the ESE stockroom. Once issued from the stockroom to a department, the caustic reagents are kept in safety carriers and stored along with carriers in the department's cabinet designated for caustics only. Note: Caustic reagents are stored in separate cabinets from the acids.
Other reagents	Stored in the main chemical or standards storage room, or stored in the designated cabinets in each department. Liquids in quantities of one gallon or more must be kept in safety carriers. Standards that require storage at 4°C or at 0°C are stored in each department's refrigerators or freezers (respectively) designated for standards only.

Source: ESE.

## 7.0 SAMPLE CUSTODY

### 7.1 SAMPLE CUSTODY OBJECTIVES

The primary objective of sample custody is to create an accurate written verified record that can be used to trace the possession and handling of the samples from the moment of collection until receipt by the laboratory. Adequate sample custody in the laboratory will be achieved by means of approved laboratory documentation.

#### 7.1.1 DEFINITION OF LEGAL CHAIN OF CUSTODY

A sample for this project is defined to be in someone's custody if:

1. It is in one's actual physical possession;
2. It is in one's view, after being in one's physical possession;
3. It is in one's physical possession and then locked or otherwise sealed so that tampering will be evident; or
4. It is kept in a secure area, restricted to authorized personnel only.

#### 7.1.2 LEGAL CUSTODY PROCEDURES

1. Formal chain of custody starts when the pre-cleaned sample containers are dispatched to the field. The sample kit preparation personnel initiate custody of the sample containers by completing the first line under the "Relinquish By" of the chain-of-custody logsheet (Figure 7-5). Receipt of the sample containers are acknowledged by the field personnel by signing and dating the first line under the "Received By" of the logsheet.
2. The formal chain of custody is signed by the Laboratory Coordinator in the laboratory. At the field, the Field Team Leader or his designate, is responsible to ensure that the chain-of-custody form is maintained.
3. Copies of the chain-of-custody form and/or field sheets are maintained with project records.
4. Errors in all documents are corrected by drawing one line through the error, then signing, and dating the corrections.
5. All documentation/logs are signed/initialed by appropriate personnel.

Due to evidentiary nature of the samples collected, possession must be traceable from the time the precleaned containers leave the laboratory to the field. Field chain of custody actually begins at the laboratory. Sample kits, which refer to the coolers, sample containers, preservatives, and trip blanks, are requested from the kit preparation staff using the Sample Kit Request Form (Figure 7-1). This form is completed by the Laboratory Coordinator and accompanied by the field group logsheets, labels, and any other relevant information. Shipping labels and/or the ESE Shipping Request Form (87201.A) (Figure 7-2) are provided in accordance with current corporate policy on sample kit handling.

The preservatives are packed in fiberboard boxes filled with vermiculites (inert materials compatible with both acids and alkalis) and labeled showing type of preservatives used.

The sample containers, boxed preservatives, trip blanks, if needed, chain-of-custody field logsheet, and a copy of the sample fraction codes are packed in coolers, sealed and shipped to the field by bonded carrier (i.e., UPS or Federal Express). All kit request forms are signed and dated upon completion by kit preparation staff. The number of coolers shipped to the field is documented in the shipping receipts that are kept in a central file located in the Gainesville, Florida, ESE Accounting office. Coolers that are picked up by the field personnel are logged out from the sample kit preparation staff using the Kit Pick-up Log in Figure 7-3. An ESE cooler tracking report indicating the personnel who prepared the kits, cooler number(s), project name and number, contents of each cooler, and the time and date the cooler was released is generated. This report is attached to the kit request form, a copy is packed in the cooler along with the other documentation, and the original filed by the kit preparation staff for future reference. An example of the cooler tracking report is shown in Figure 7-4.

SAMPLE KIT PREP & SHIPPING REQUEST FORM

FROM \_\_\_\_\_ EST. # \_\_\_\_\_

PROJECT NAME \_\_\_\_\_

FIELD GROUP (I) / SAMPLE # (II) \_\_\_\_\_

Submitted to Kit Prep on \_\_\_\_\_ for shipment between \_\_\_\_\_ and \_\_\_\_\_

Kit completed by \_\_\_\_\_ on \_\_\_\_\_

Transferred to \_\_\_\_\_ Facility by \_\_\_\_\_ on \_\_\_\_\_

TRIP BLANKS? H/T, # OF, Y? \_\_\_\_\_ Y \_\_\_\_\_ ED \_\_\_\_\_ Ovr \_\_\_\_\_

PRESERVATIVES/FIELD SUPPLIES & EQUIPMENT TO BE SHIPPED \_\_\_\_\_ in KETCHU \_\_\_\_\_ in Sealed Container

TYPE AND # OF REQUIRED PRESERVATIVE BOTTLES (25mL PER BOTTLE)

For CH (2 in dist): \_\_\_\_\_ Cadmium Nitrate (per pot 5.1 per ml): \_\_\_\_\_ Lead Acetate (per 100 ml 5.1)

Hydrochloric \_\_\_\_\_ HCl (per M.R.) Sodium Hydroxide (per B.M.) Sodium Sulfate (per TORCH 2 samples)

Sodium Thiosulfate (per TRIM - CH's samples) \_\_\_\_\_ Sulfate (per S.O.D.) Zinc Acetate (per M)

OTHER SUPPLIES \_\_\_\_\_ Glass \_\_\_\_\_ Y. Labels \_\_\_\_\_ Potenti \_\_\_\_\_ Y. Train Bags (for kit)

\_\_\_\_\_ pH Scales \_\_\_\_\_ Evidence Tags \_\_\_\_\_ Other \_\_\_\_\_

FOR PICKUP BY \_\_\_\_\_ or \_\_\_\_\_ SHIP AS FOLLOWS:

TO: _____	WEIGHT/VOLUME
Name _____	PER PACKAGE
Company _____	
Street Address to UPS, Fed Ex (NO P.O. Boxes) _____	1. _____ / _____ 10. _____ / _____
City _____ State _____ Zip _____	2. _____ / _____ 11. _____ / _____
Phone # _____	3. _____ / _____ 12. _____ / _____
Printed # for Shipping _____	4. _____ / _____ 13. _____ / _____

SERVICE REQUIRED

Special Service Request?  Y

Airborne  DCL  Re-Overpack

Fed Express  Next AM  Next PM  2-Day/OD

Tracking  Add Container  Contents (not of same org)

UPS:  Next Day  2-Day  Std (1-2 day in Air)

USPS:  Exped Service  First Class  Other \_\_\_\_\_

Box: type number? \_\_\_\_\_  Other \_\_\_\_\_

Kit shipped by \_\_\_\_\_ on \_\_\_\_\_ to arrive \_\_\_\_\_ on \_\_\_\_\_ by \_\_\_\_\_ AM \_\_\_\_\_ PM

OTHER NOTES

Figure 7-1  
SAMPLE KIT PREP & SHIPPING  
REQUEST FORM

SOURCE: ESE

ENVIRONMENTAL SCIENCE  
& ENGINEERING, INC.

ESE SHIPPING REQUEST

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

FROM \_\_\_\_\_ EXT. \_\_\_\_\_

PROJECT/PROPOSAL/CHARGE NO.: \_\_\_\_\_

TO \_\_\_\_\_

Name \_\_\_\_\_

Company \_\_\_\_\_

Street Address for UPS, Federal Express (NO P.O. Boxes) \_\_\_\_\_

City \_\_\_\_\_ State \_\_\_\_\_ Zip \_\_\_\_\_

( ) \_\_\_\_\_

Telephone \_\_\_\_\_

DELIVERY TIME: PACKAGE MUST REACH DESTINATION BY:

\_\_\_\_\_ Day \_\_\_\_\_ Time \_\_\_\_\_

SATURDAY SERVICE REQUIRED

DECLARED VALUE: \_\_\_\_\_

WEIGHT (for more than one package, list each one) \_\_\_\_\_

TYPE OF SERVICE REQUIRED

- |   |   |
|---|---|
| <input type="checkbox"/> 1st CLASS MAIL | <input type="checkbox"/> FEDERAL EXPRESS 2-DAY    |
| <input type="checkbox"/> UPS GROUND     | <input type="checkbox"/> FEDERAL EXPRESS NEXT DAY |
| <input type="checkbox"/> UPS 2-DAY      | <input type="checkbox"/> DHL                      |
| <input type="checkbox"/> UPS NEXT DAY   | <input type="checkbox"/> OTHER _____              |

FORM 87201A

Figure 7-2  
ESE SHIPPING REQUEST FORM

SOURCE: ESE.

ENVIRONMENTAL SCIENCE  
& ENGINEERING, INC.





05/17/93

Environmental Science & Engineering, Inc. 12-23-92 \*\*\* FIELD LOGSHEET \*\*\* FIELD GROUP: EXAMPLE  
 PROJECT NUMBER J924000V 0000 PROJECT NAME: COMPANY XXX LAB COORD. PORTIA FISGAN

USE #	SITE/STA HAZ?	FRACTIONS(CIRCLE)	DATE	TIME	LAB COORD.
*1	HW-1	EC EC VP VP VP			WG10LC
*2	HW-2	EC EC VP VP VP			WG10LC
*3	HW-3	EC EC VP VP VP			WG10LC
*4	HW-4	EC EC VP VP VP			WG10LC
*5	HW-5	EC EC VP VP VP			WG10LC
*6	HW-6	EC EC VP VP VP			WG10LC
*7	HW-7	EC EC VP VP VP			WG10LC
*8	HW-8	EC EC VP VP VP			WG10LC
*9	HW-9	EC EC VP VP VP			WG10LC
*10	HW-10	EC EC VP VP VP			WG10LC

NOTE -CHANGE OR ENTER SITE ID AS NECESSARY; UP TO 9 ALPHANUMERIC CHARACTERS MAY BE USED  
 -CIRCLE FRACTIONS COLLECTED. ENTER DATE, TIME, FIELD DATA (IF REQUIRED), HAZARD CODE AND NOTES  
 -HAZARD CODES: I-INSTANT C-COMPOUND R-REACTIVE T-TOXIC WITH H-OTHER HAZARD: IDENTIFY SPECIFICS IF KNOWN  
 -PLEASE RETURN COMPLETED LOGSHEETS WITH SAMPLES TO Environmental Science & Engineering, Inc.

RELINQUISHED BY: (NAME/ORGANIZATION/DATE/TIME) VIA: REC'D BY (NAME/ORGANIZATION/DATE/TIME)  
 1 \_\_\_\_\_  
 2 \_\_\_\_\_  
 3 \_\_\_\_\_

SAMPLER: Shipped on Ice? Yes/No; I anticipate shipping      (1) more samples on     /      
 SAMPLE CUSTODIAN: Custody Seals Used? Yes/No; If Yes, Seals Intact? Yes/No Interior Temp?      Deg C  
 Preservatives Audited? Yes/No Any Problems? Yes/No; If Yes, describe:     

Figure 7-5  
 CHAIN-OF-CUSTODY FIELD LOGSHEET

SOURCE: ESE

ENVIRONMENTAL SCIENCE  
 & ENGINEERING, INC.

### 7.1.3 DOCUMENTATION

The records for laboratory sample custody include:

#### 1. Laboratory Forms:

- Sample Kit Request Form (Figure 7-1),
  - ESE Shipping Request Form (Figure 7-2),
  - Kit Pick-Up Log (Figure 7-3),
  - Example of Cooler Tracking Report (Figure 7-4),
  - Sample Label (Figure 7-6),
  - Standardized Sample Preservation Codes (Figure 7-7),
  - Sample Chest Custody Form (Figure 7-8),
  - Cold Room Sample Arrival Logbook (Figure 7-9),
  - Sample Check In/Out Log (Figure 7-10),
  - VOA GC Sample Thru-Log (Figure 7-11),
  - VOA GC/MS Sample Thru-Log (Figure 7-12),
  - Radiochemistry Sample Storage and Custody Logsheet (Figure 7-13),
  - Conductivity Meter Calibration Form (Section 9.0), and
  - pH Meter Calibration Form (Section 9.0).
2. Sample Extraction Log (Organic Laboratory/Extraction Logsheet, Figure 7-14).

Errors in all documents are corrected by following the procedure in Section 7.1.2.

### 7.2 LABORATORY CUSTODY

Sample chests (packages/coolers) are transported to the laboratory. The deliverer will sign, date, and indicate the time of delivery, the number of packages, the Laboratory Coordinator or addressee, and any comments including visible or suspected physical condition of the packages into the Sample Chest Custody Logbook (Figure 7-8). The chests are then recorded as having been received by the laboratory in the Sample Chest Custody Logbook by the Sample Custodian.

FBI 3742DDY DDD MW-10  
COMPANY XXX

EXAMPLE-10-VP

SAMPLER DATE

COND PH

TIME

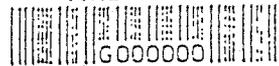


Figure 7-6.  
SAMPLE LABEL

SOURCE: ESE

ENVIRONMENTAL SCIENCE  
& ENGINEERING, INC.













Field Group	Sample Number	Delivery Date / Time	Check Out	Check In	Purpose of Removal
			Time / Date	Time / Date	
			Analyst	Analyst	
			Time / Date	Time / Date	
			Analyst	Analyst	
			Time / Date	Time / Date	
			Analyst	Analyst	
			Time / Date	Time / Date	
			Analyst	Analyst	
			Time / Date	Time / Date	
			Analyst	Analyst	
			Time / Date	Time / Date	
			Analyst	Analyst	
			Time / Date	Time / Date	
			Analyst	Analyst	
			Time / Date	Time / Date	
			Analyst	Analyst	
			Time / Date	Time / Date	
			Analyst	Analyst	

Figure 7-13  
 RADIOCHEMISTRY SAMPLE STORAGE  
 AND CUSTODY LOG SHEET

SOURCE: ESE.

ENVIRONMENTAL SCIENCE  
 & ENGINEERING, INC.



The samples are checked in by the Sample Custodian for proper preservation (e.g., pH, temperature), integrity (e.g., leaking, broken bottles), and proper and complete sample documentation and ID. Sample chests or coolers that are not within the  $4 \pm 2$  degrees Celsius ( $^{\circ}\text{C}$ ) requirement are reported immediately to the Laboratory Coordinator to determine if resampling will be required. All samples contained in the shipment are compared to the logsheet(s) to ensure that all samples designated on the logsheet have been received. Any changes in station ID from the originally established station ID are noted. The Sample Custodian will note any special remarks concerning the shipment. The Sample Custodian reviews the integrity of all sample fraction containers, checks the accuracy and clarity of all documentation received, and scans all the samples for radioactivity level. The Sample Custodian audits all fractions requiring field preservation to ensure that they have been properly preserved. The Sample Custodian will preserve unpreserved fractions or add additional preservative, if needed, on receipt. Deficiencies in sample preservation, additional preservative added, and all other inadequacies are recorded on the logsheet and reported to the Laboratory Coordinator.

All insufficiencies and/or discrepancies are recorded on the logsheet and immediately reported to the appropriate Laboratory Coordinator. The Laboratory Coordinator will inform the Project Manager and field team. The Project Manager, upon consultation with the Project QA Coordinator, may decide if resampling is required.

After all samples and documentation have been reviewed and appropriately annotated, the Sample Custodian signs the logsheet and submits it to Information Services for processing. Any marks or notes made on the chain-of-custody document by the Sample Custodian should be clearly distinguished from original field notations.

Shipping receipts are stapled on chain-of-custody logsheets and stored in the project file.

Samples are placed in appropriate storage areas in the laboratory depending on storage requirements. The Department Managers or their designee are notified that the samples have arrived through the distribution of arrival notices. The majority of the samples are

stored in the main coldroom with the temperature maintained at  $4 \pm 2^{\circ}\text{C}$ . The Sample Custodian will log the samples delivered into the coldroom in the Cold Room Sample Arrival Logbook (Figure 7-9). The coldroom is kept locked when not in use. The water samples for metals analysis (fractions N and NF) are stored in a separate air-conditioned storage room located near the metals sample preparation area. This room is also kept locked if not being used by the analyst. The samples in these storage areas are assigned to labeled shelves by field group. A sample location list is posted at the door of each storage room. Access to samples is limited to authorized personnel, and a Sample Check In/Out Log is maintained (Figure 7-10).

The samples for volatile organics are delivered directly to the gas chromatography (GC)-Volatiles or gas chromatography/mass spectrometry (GC/MS) Department by the Sample Custodian and are stored in the department's refrigerators designated for sample storage only. The samples delivered to these departments by the Sample Custodian are logged in the department's Sample Thru-Log (Figures 7-11 and 7-12). Sample fractions for volatiles are stored separate from standards or semivolatile fractions in order to minimize cross-contamination. Samples remain in storage until it is unnecessary to retain them, at which time their disposition (in accordance with Section 8.0) is noted.

The samples for radiochemistry analyses are delivered directly to the Radiochemistry Department and logged into the Sample Storage and Custody Logsheet (Figure 7-13). Samples checked out and checked in from this storage room are also documented in the Sample Storage and Custody Logsheet.

When it is necessary to use another laboratory for sample analysis, the Laboratory Coordinator is responsible for arrangements with the second laboratory. The samples will only be subcontracted to a NEESA, HAZWRAP, FDER, state and federal government agency, or client-approved laboratory (as appropriate). Specific NEESA/HAZWRAP approval is required prior to subcontracting. Documentation to transfer to another laboratory must include: collection data and time, field ID, laboratory ID, date of sample preparation, and requested analyses.

The samples should be chilled prior to and during shipment. A logsheet indicating samples and fractions sent must accompany the samples to the subcontractor. The subcontractor should sign and date the logsheet upon receipt of the samples. A copy of the signed logsheet will be returned to ESE and placed in the project file.

### 7.3 LABORATORY INFORMATION MANAGEMENT SYSTEM (LIMS)

CLASS™ is an automated, inhouse-developed LIMS that integrates information from sample collection, laboratory analyses, and QC requirements; and calculates, checks, stores, and reports data in a variety of formats. CLASS™ resides on a Novell Arcnet (using Novell SFT, version 2.11) IBM-PC-compatible network with 1,600 megabytes of storage and is connected to more than 80 personal computers (PCs) throughout the Gainesville chemistry laboratories and offices. CLASS™ is managed by the Laboratory Information Services Department within the Gainesville laboratory. All data from analyses performed by the laboratory are managed and stored using CLASS™.

The database is stored, processed, and retrieved using the database manager Advanced Revelation® (copyright COSMOS, Inc.). The file structure and indexing provided by Advanced Revelation® allow easy retrieval, grouping, and formatting of data. Incorporated into the system is the ability to combine field data, analytical results, and QC data and produce specially formatted project-specific reports, statistical analyses, plots, and electronic files.

CLASS™ manages the flow of samples and data through the laboratory. Prior to sampling, the Laboratory Coordinator provides information on the number of samples, site IDs, parameters to be analyzed, and estimated collection dates. This information is entered into CLASS™ and used to produce sample labels and chain-of-custody logsheets (Figure 7-5). A unique ESE number is assigned to each sample, and labels with that number and the site ID are placed on each container for that sample. At each site, samples are collected and placed in the appropriate pre-labeled containers. Sampling information is recorded on the field logsheet. Samples accompanied by the field logsheet

are sent to the laboratory where they are checked and processed by the Sample Custodian. Samples are stored in the coldroom at  $4 \pm 2^{\circ}\text{C}$ . The logsheet is submitted to the Laboratory Information Services Department, where the samples, along with the date of collection and site ID, are logged into CLASS™. Logsheets are placed in the project file and maintained by Information Services.

ESE uses a combination of EPA Storage and Retrieval (STORET) numbers and company-assigned Method Codes to designate parameters required for analysis. Each STORET-method combination has its own laboratory QC requirements specific to that analytical method stored in CLASS™. A list of all required parameters is logged into the computer with each sample. This list is identified on the field logsheet for each sample.

The sampling information is entered into the computer to activate the parameter list for the samples collected and received by the laboratory. A report (Available Numbers) of samples available for each analysis indicates the number of days left before the holding time is exceeded for each method for each sample. This report is distributed daily to each analytical department, and the information also can be accessed readily from CLASS™ by the Laboratory Coordinator or any analyst in the laboratory.

CLASS™ uses a batch method for analyzing, checking QC, and calculating final results of samples. Prior to analyzing a sample batch, the analyst will designate a specified group of samples in the computer and the sample-parameter status will be updated to "IL" ("In Laboratory"). The analytical batch is assigned a unique batch control number, which is stored with all final data, to facilitate data review, QC reporting, and retrieval of original documentation.

The production of each laboratory batch usually requires several distinct activities. Instrument calibrations are entered first and includes several QC checks by CLASS™. The linear (or quadratic or logarithmic) regression equation and correlation coefficient are calculated from the calibration curve data, and the correlation coefficient is tested to determine whether it is within an acceptable range specific to the analysis. Method blank

and control spike information are then entered, and results are calculated and checked against control limits for that method. Sample responses are entered into the batch, and final concentrations are calculated for each sample. Responses are checked to ensure that they are bracketed by the standard curve. The batch printout includes a QC summary showing the automated QC checks, such as holding times, the presence of spikes, and acceptable spike recoveries. Any discrepancies are flagged by the computer for the analyst.

The batch printout also documents that the analyst has checked data entries and provided all required documentation for the analysis. The batch printout is completed, signed, and dated by the analyst, and reviewed and signed by the Department Manager or a designated reviewer.

Analysts use the PC to reserve samples for analysis, calculate final concentrations, and interactively check calibration curves and QC results. By allowing the analyst to enter data directly and check QC and sample results, the analyst is made aware of QC problems. When the analyst has entered the curve and QC and sample data, the batch printout (including checklists), worksheets, copies of notebook pages and any other pertinent documentation (e.g., chromatograms), are placed in a file folder with the assigned batch number and submitted to Information Services. Information Services personnel process the batch in the computer to verify QC and to update the sample records and final calculated concentrations.

Each employee is assigned an individual access code for entry into CLASS™. Laboratory personnel and Laboratory Coordinators are not permitted to update sample records; this is done exclusively by Information Services. All personnel with an access code may retrieve information from the system.

Once a batch has been finalized by Information Services, the batch is locked, and data cannot be changed by the analyst. If a data change is necessary, a Batch Update Request Form (Figure 7-15) must be completed. This form requires the reason for the change.

BATCH UPDATE

Batch # \_\_\_\_\_ Analysis \_\_\_\_\_ Dept. # \_\_\_\_\_

Request to (circle one):      DEFINALIZE      or      CHANGE

Initiated by: \_\_\_\_\_ In Order to: \_\_\_\_\_

APPROVED/ACKNOWLEDGED BY:

Dept. Manager \_\_\_\_\_ Date \_\_\_\_\_

Lab Coordinator(s) \_\_\_\_\_

\_\_\_\_\_

\_\_\_\_\_

Changes Processed by: \_\_\_\_\_

UPDATED Batch Reviewed & Approved by: \_\_\_\_\_

Batch Refinalized by: \_\_\_\_\_

Figure 7-15  
BATCH UPDATE REQUEST FORM

SOURCE: ESE.

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the approval of the Laboratory Department Manager, and the approval of the Laboratory Coordinator. The form is then submitted to Information Services, where the original batch folder is retrieved. Information Services personnel make the change, the form is stapled to the new batch printout, and the updated batch is reviewed and approved by the Laboratory Department Manager and Laboratory Coordinator.

The batch folders, with all supporting documentation, are filed chronologically by department in a secured Information Services storage room; file cabinets with project files are stored similarly. These may be signed out for review by the analysts, Laboratory Coordinators, or QA personnel. A program in CLASS™ is used to track folders that have been checked out. Batch folders and project files are kept a minimum of 5 years.

Laboratory personnel and Laboratory Coordinators use the computer to monitor the flow of data through the system. Data are accessed and reported by sampling event, project, or any subset of samples and parameters. A log of electronic transfers is kept by Information Services. CLASS™ enables the Laboratory Coordinator to:

1. Produce a variety of summary reports of analytical data;
2. Calculate parameters from analytical data (e.g., cation/anion balance);
3. Calculate statistics such as mean, maximum, minimum, and standard deviation;
4. Calculate atmospheric concentrations;
5. Produce a data file to be read into the Statistical Analysis System (SAS) for in-depth statistical analysis;
6. Summarize QC in various formats; and
7. Produce a project-specific export-data file.

Data are stored in the CLASS™ database and can be exported electronically into Lotus and DBASE files. Many client-requested formats have been developed in CLASS™ for electronic data transfer. When a client requests an electronic data transfer, a

regularhardcopy data report is usually sent in addition to the electronic file. Copies of both electronic and hard copies are maintained in project files.

Information Services has a staff of computer programmers to maintain and modify CLASS™. Requests for new programs or changes are kept in both electronic and hardcopy files; the names of the person making the request and the programmer are included. Every change made to a program is documented electronically at the end of the program with the date, employee number of the programmer, and a brief description of the change. A summary of these changes is maintained in CLASS™ listing the programs, changes, requestors, and programmers. All program revisions are documented in a revisions file and can be reviewed anytime. Completed requests are tested by the programmer staff and then verified by the requestor.

The Laboratory QC Coordinator validates a portion of the data quarterly by recalculations from the raw values and verification that the computer is performing calculations correctly.

The QA Division routinely checks data packages including computer printouts to verify that CLASS™ data match raw data from the laboratory.

The database is backed up daily except Saturday using optical disks or equivalent high-density storage media. These disks are stored in the Information Services air-conditioned locked storage room located in a separate building. Archived electronic data are stored in special files accessible by Information Services personnel. Laboratory Coordinators can access the status of all data (including archived samples in CLASS™) and may request that sample data be restored for more thorough review.

## 8.0 ANALYTICAL PROCEDURES

### 8.1 STANDARD PROCEDURES

Standard analytical procedures to be used for any project for chemical analysis of water and soil are referenced in Section 5.0. Laboratory Department Managers will ensure that only these standard analytical methods are employed by the staff. Standard analytical methods manuals are required for all departments, and development of the documents are ultimately the responsibility of the department managers. The methods cited in these documents will be the methods normally used. Any deviation from the standard method must be documented in the analyst notebook and approved by the department manager.

For parameters not listed, nonstandard methods may be specified by the client or developed by the laboratory. Nonstandard methods must be validated as described in Section 8.3.

### 8.2 NONSTANDARD METHODS VALIDATION

If other than standard analytical methods become necessary due to a change in work scope, it is necessary to validate the analytical method. The responsible department manager must establish thorough method validation so that the method selected measures the reported parameter with the necessary precision, accuracy, and detection limit and without severe interference by other constituents in the sample. Major modifications of standard methods such as extraction, preparation, and cleanup procedures and/or the application of a standard method to new analytes or matrices will require method validation. Nonstandard methods will be submitted to and state or government agency (i.e. FDER, USACE, etc) and client, if required, FDER for review and approval prior to use on samples for analyses.

The following subsections constitute the minimum requirements for initial establishment of the accuracy, precision, and detection limit of nonstandard methods.

For each parameter of interest, seven replicate spike samples will be prepared from laboratory blank water (for water methods) or an uncontaminated "standard" appropriate matrix (for soil or tissue methods) at one appropriate analyte concentration. Spiked samples will be analyzed according to the method. An unspiked "standard" matrix blank or unspiked laboratory blank water will be analyzed. The spiking concentration should be selected such that the final extract or aliquot can be analyzed with less than tenfold dilution in the midrange of the calibration curve.

The detection limit of each parameter of interest will be determined according to the protocols described in 40 CFR Part 136 Appendix B.

Accuracy (Recovery)--The minimum requirements for initial establishment of accuracy for nonstandard methods are as follows:

1. Calculate the found concentration for each spiked sample as follows:  
 $R = \text{measured concentration} = \text{measured concentration in spiked sample} - \text{measured concentration in unspiked (blank) sample}.$
2. Calculate the percent recovery for each spiked sample as follows:

$$P = \frac{R}{S} \times 100\%$$

where:  $R$  = measured concentration for each spiked sample, and  
 $S$  = target concentration for each spiked sample.

3. Calculate the average percent recovery and relative standard deviation of the percent recovery for the spiked "standard" samples as follows:

$$\bar{P} = \frac{P_1 + P_2 + P_3}{3} = \text{average percent recovery (standard samples)}$$

where:  $S_p$  = standard deviation of  $P$

$$S_r = \sqrt{\frac{1}{n-1} \left[ \left( \sum_{i=1}^n R_i^2 \right) - \frac{1}{n} \left( \sum_{i=1}^n R_i \right)^2 \right]}$$

where: n = number of recovery values, and  
RS<sub>r</sub> = relative standard deviation of P.

$$RS_r = \frac{S_r}{P} \times 100\%$$

Precision--The minimum requirements for initial establishment of precision for nonstandard methods are as follows:

1. Calculate the RPD between each pair of replicate sample matrix spike samples.

$$RPD_1 = \frac{|R_1 - R_2|}{(R_1 + R_2)/2} \times 100$$

$$RPD_2 = \frac{|R_1 - R_3|}{(R_1 + R_3)/2} \times 100$$

$$RPD_3 = \frac{|R_2 - R_3|}{(R_2 + R_3)/2} \times 100$$

2. Calculate the average RPD for the sample matrix spikes.

$$RPD = \frac{RPD_1 + RPD_2 + RPD_3}{3}$$

Detection Limit--The detection limit of the method is the lowest sample concentration that can be reliably recovered and measured in the sample matrix with a low background level. Statistically based procedures to determine absolute method detection limits (MDLs) as described in 40 CFR Part 136 Appendix B will be used. The reported detection limit for a method will be subject to the judgment of the analyst and the department manager and should take into account background levels, instrument baseline noise, spiking recoveries, and the lowest calibration standards analyzed. In general (except for those methods where the detection limit is derived from instrument considerations), the reported detection limit for a method is determined by the lowest standard concentration analyzed, taking into consideration the sample volume or weight of sample used and the final extract volume (where applicable).

Method validation and method detection limits determination results should be recorded and submitted to the Department Manager and Project QA Supervisor (if specified) or Laboratory QA/QC Manager prior to the initiation of analysis. Before analysis begins, the department manager will assure that the method meets the performance criteria required by the project.

Once the method is validated, these initial validation data (precision and accuracy) are periodically revised, updated, and improved using the data acquired during the laboratory's routine analytical QC program.

### 8.3 LABORATORY GLASSWARE

Dirty glasswares are drained of solvents and rinsed with tap water. If soils or other residues are still remaining, before they are submitted to the ESE washroom for cleaning. Glasswares from the Metals and Radiochemistry Departments are always rinsed with tap water prior to submittal to the washroom.

A completed Glassware Washing Request Form (Figure 8-1) must accompany each box of glasswares brought to the ESE washroom. All laboratory glasswares (i.e., volumetric flasks, separatory funnels, extraction tubes, beakers, graduated cylinders, and others) are cleaned according to the analysis/parameter group listed in Table 8-1. These cleaning procedures are subject to change depending on the requirements of the projects. The washroom personnel perform cleaning procedures 1 through 4 listed in the table, unless otherwise directed in writing by the analyst via the Glassware Washing Request Form. Cleaned glassware for organic analyses are placed in boxes lined with fresh aluminum foil. The form is then initialed, dated, and the type of cleaning procedures performed specified by the washroom personnel. The remaining cleaning procedures are performed by the analyst.

GLASSWARE WASHING REQUEST FORM

TO BE DONE

Normal Wash 1) Hot soapy tap water wash  
2) Tap water rinse  
3) DI rinse

Rinse with DI only!

Other \_\_\_\_\_ (be specific)

\_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_

SOLVENT RINSE

Acetone

Other \_\_\_\_\_

\_\_\_\_\_  
\_\_\_\_\_

NEEDED BY: \_\_\_\_\_  
Date and Time

SPECIAL INSTRUCTIONS: \_\_\_\_\_

\_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_

REQUESTED BY: \_\_\_\_\_

THE FOLLOWING HAS BEEN COMPLETED

Normal Wash

Rinse with DI only!

Other \_\_\_\_\_

\_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_

SOLVENT RINSE

Acetone

Other \_\_\_\_\_

\_\_\_\_\_  
\_\_\_\_\_

COMPLETED BY \_\_\_\_\_

DATE \_\_\_\_\_

Figure 8-1  
GLASSWARE WASHING  
REQUEST FORM

SOURCE: ESE.

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Table 8-1. Glassware Cleaning Procedures

Analysis/Parameter	Cleaning Protocol*
Extractable Organics	1.2.3.4.5.6
Purgeable Organics (Volatiles)	1.2.3.4.7.13
HPLC Analyses	1.2.3.4.5.10
EDB, DBCP, THMS	1.2.3.4.5.8.13
Trace Metals	1.2.3.4.12
Nutrients	1.2.3.4.11
Minerals	1.2.3.4
Residues	1.2.3.4.14
Cyanide, Oil and Grease, Phenols	1.2.3.4
Petroleum Hydrocarbons	1.2.3.4.5.9
COD, BOD	1.2.3.4
Radiochemistry	1.2.3.4

Note: DBCP = 1,2-Dibromo-3-chloropropane  
 EDB = 1,2-Dibromoethane  
 HCL = Hydrochloric acid  
 HNO3 = Nitric acid  
 HPLC = High Pressure Liquid Chromatography  
 THMS = Trihalomethanes

\*Cleaning Procedures

1. Remove all labels using sponge or acetone.
2. Wash with hot soapy water (use Liuminox soap only) using brushes to scrub inside of glasswares, stopcocks, and other small pieces if possible.
3. Rinse three times with hot tap water.
4. Rinse three times with deionized water.
5. Rinse thoroughly with pesticide grade acetone.
6. Rinse with pesticide grade Methylene Chloride.
7. Rinse with pesticide grade methanol.
8. Rinse with pesticide grade hexane.
9. Rinse with appropriate extraction solvent prior to use.
10. Rinse with pesticide grade acetonitrile and pesticide grade methanol prior to use, if needed.
11. Acid rinse with 1:1 HCL, using only metals grade HCL.
12. Acid rinse with dilute HNO3 and then with deionized water prior to use.
13. Bake at 80°C for 3-4 hours.\*
14. Bake at 130°C for 3-4 hours.\*

\*Class A volumetric glassware should not be baked.

Source: ESE.

#### 8.4 LABORATORY METHOD MODIFICATIONS

Laboratory method modifications are done either to improve the method efficiency, apply a water method with the addition of appropriate sample preparation/digestion method to soils or add new compounds to an approved method. ESE has several method modifications involving the addition of new compounds to a specific EPA method(s) or applying a water method with the addition of appropriate sample digestion/extraction method to soils. These compounds are listed in Table 8-2 and their QA targets are found in Section 5.0. Method validation have been performed and the method validation packages were submitted to FDER for approval. These method validation studies were referenced in the appendices of this LCQAP. The method validation packages include precision and accuracy data, method detection limit studies, copy of the method used, and raw data. Method validation were performed on the following compounds listing the method and matrix used:

Table 8-2. Laboratory Method Modification Compounds

Compound/Parameter	EPA Method	Matrix
Cyanide	EPA 9010	Soil
Total Organic Carbon	EPA 9060	Soil
Hexavalent Chromium	EPA 3060/7196	Soil
Total Petroleum Hydrocarbons (TRPH)	EPA 3550, 415.1	Soil
TOX	EPA 9020	Soil
Gross Alpha	EPA 3050, 9310	Soil
Gross Beta	EPA 3050, 9310	Soil
Radium 226	EPA 3050, 9320	Soil
Radium 226, alpha emitters	EPA 3050, 9315	Soil
Radium 228	EPA 3050, 9320	Soil
Freon 113	EPA 8010	Water
MTBE	EPA 8010	Water
Isodrin	EPA 8080	Water & Soil
Kepon	EPA 8080	Water & Soil
Metolchlor	EPA 8080	Water & Soil
Kelthane (Dicofol)	EPA 8080	Water & Soil
Aachlor	EPA 8140	Soil
Metribuzin	EPA 8140	Soil
EPTC	EPA 8140	Water & Soil
Butylate	EPA 8140	Water & Soil
Pebulate	EPA 8140	Water & Soil
Vernolate	EPA 8140	Water & Soil
Atrazine	EPA 8140	Water & Soil
Terbutol	EPA 8140	Water & Soil
Hexazinone	EPA 8140	Water & Soil
Famphur	EPA 8140	Water & Soil
O,O,O-Triethyl Phosphorothionate	EPA 8140	Water & Soil
Sulfoapp	EPA 8140	Water & Soil
Thionazin	EPA 8140	Water & Soil
Methylethyl ketone (MEK)	EPA 8240	Water & Soil
Methylisobutyl ketone (MIBK)	EPA 8240	Water & Soil
Acrolein	EPA 8240	Water & Soil
Acrylonitrile	EPA 8240	Water & Soil

Table 3-2. Laboratory Method Modification Compounds  
 (Continued, Page 2 of 2)

Compound/Parameter	EPA Method	Matrix
Carbon disulfide	EPA 8240	Water & Soil
Acetophenone	EPA 8270	Water & Soil
4-Aminobiphenyl	EPA 8270	Water & Soil
4-Chloroaniline	EPA 8270	Water & Soil
2,6-Dichlorophenol	EPA 8270	Water & Soil
p-(Dimethylamino)azobenzene	EPA 8270	Water & Soil
7,12-Dimethylbenz(a)anthracene	EPA 8270	Water & Soil
Ethyl methanesulfonate	EPA 8270	Water & Soil
o-Cresol	EPA 8270	Water & Soil
p-Cresol	EPA 8270	Water & Soil
1-Naphthylamine	EPA 8270	Water & Soil
2-Nitroaniline	EPA 8270	Water & Soil
3-Nitroaniline	EPA 8270	Water & Soil
4-Nitroaniline	EPA 8270	Water & Soil
N-Nitroso-di-n-butylamine	EPA 8270	Water & Soil
N-Nitrosomethylethylamine	EPA 8270	Water & Soil
N-Nitrosopiperidine	EPA 8270	Water & Soil
Pentachlorobenzene	EPA 8270	Water & Soil
Pentachloronitrobenzene	EPA 8270	Water & Soil
Phenacetin	EPA 8170	Water & Soil
2-Picoline	EPA 8270	Water & Soil
Pronamide	EPA 8270	Water & Soil
1,2,4,5-Tetrachlorobenzene	EPA 8270	Water & Soil
2,3,4,6-Tetrachlorophenol	EPA 8270	Water & Soil
2,4,5-Trichlorophenol	EPA 8270	Water & Soil
1,3,5-Trichlorobenzene	EPA 8270	Water & Soil

### 8.5 REAGENT STORAGE

The procedures for storing reagents in the laboratory are presented in (Section 6.0). All reagents are marked with date received and date opened.

### 8.6 LABORATORY WASTE DISPOSAL

It is important that all waste materials generated in the laboratory be disposed of promptly and properly. The following subsections describe the procedures for handling laboratory wastes.

#### 8.6.1 LIQUID WASTES

1. In general, no chemical wastes may be disposed of in the sinks.
2. Only certain dilute acid wastes can be disposed of in the sinks.
3. Disposal of Standards and Solutions--As standards and solutions are made, the solvent, constituents, date, and initials must be put on the container. This information must be on the container before it is offered for disposal. Standards containing any amount of organic solvents must not be poured down the sink. Aqueous standards of organic or inorganic (metals, etc.) compounds must either be disposed of in the appropriate waste drum, or picked up by the waste technician.
4. Disposal of Solvent Wastes--All waste solvents should be disposed of in the red waste-solvent containers located throughout the different departments in the laboratory. Solvents should be segregated according to the designated chemical types and placed only in the appropriate waste-solvent container. The waste containers will be emptied on a regular basis. If the containers become full before then, the hazardous materials technician should be called so the containers can be emptied.

Solvents will be segregated as follows:

<u>Freon Waste</u>	<u>Chlorinated Solvents</u>	<u>Flammable Solvents</u>		<u>HPLC Solvents</u>
Freon-112	Methylene chloride	Hexane	Benzene	Methanol
	Chloroform	Acetone	Toluene	Acetonitrile
	Carbon tetrachloride	Pentane	Nylene	Water
		Ethyl ether	Petroleum ether	Tetrahydro- furan
		Isopro- panol	Cyclohexane	Isopropanol

\*Note: Isopropanol may be disposed of in either the flammable or HPLC container.

Specially marked waste cans are available in the water quality laboratory for waste Freon-112. Freon should never be disposed of with other chlorinated solvents.

Glass jugs will not be accepted for solvent waste. Solvents must be segregated as shown and put into the red waste containers. The hazardous waste technician should be called if the solvent cans should fill during the day, and they will be emptied as soon as possible.

5. Disposal of Extracted Water Samples--Water samples which have been solvent extracted should be disposed in the extracted water waste disposal drum located in each department.

### 8.6.2 SOLID WASTES

1. Solvent saturated solids, such as sodium sulfate saturated with methylene chloride, should be disposed of in the red solid waste cans.

2. Soil samples which have been extracted with a solvent should be disposed of in the red solid waste cans in the laboratory. These containers have a closed lid to prevent solvent fumes from entering the laboratory air. Full containers will be collected by the hazardous waste technician on a regular basis. The contents of this container will be allowed to air dry and disposed of in the trash by the hazardous waste technician.
3. Disposal of Old or Contaminated Chemicals--Commercial chemicals that are out of date or contaminated should be left in their original containers. The label should be secured and the date and initials should be marked on the container. The hazardous materials technician should then be called to pick up the material.
4. Disposal of 1-milliliter (mL) and 5-mL Autosample Vials Containing Extracts-- These are to be collected in the vial collection containers in each laboratory. The containers will be emptied on a regular basis by the hazardous waste technician. If the container should become full, call the technician to have it emptied.
5. Disposal of Additional Hazardous Material--The contents will be clearly marked on the container or on an accompanying analysis report. Again, clearly date and initial the container. Contact the hazardous materials technician for pickup and disposal.
6. Disposal of Unmarked Containers, or Unknowns--These are brought to the attention of the department manager. Unknowns should not be allowed to accumulate or be misplaced. Unknowns cannot be taken for disposal until they are identified.

### 8.6.3 SAMPLE WASTES

The following procedures are employed in the disposal of excess samples that have completed all necessary testing:

1. Samples stored in the coldroom will be handled as follows:
  - a. Samples will be disposed of six weeks after the sampling date unless a longer storage time is authorized by the Laboratory Coordinator.
  - b. Prior to disposal, a sample throw-out report will be generated by the coldroom technician and taken to the Laboratory Coordinator/Project Manager for disposal authorization on each project.

- c. The Laboratory Coordinator must use the guidelines for sample disposal to determine if the sample is hazardous.
  - d. Nonhazardous soil samples will be disposed of by bulking them into a drum for offsite disposal. All labels on containers must be removed prior to disposal.
  - e. Nonhazardous water samples will be disposed by the coldroom technician in the following manner:
    - 1) Water samples will be bulked into the nonhazardous water waste tanks for offsite disposal.
    - 2) Empty sample containers should be disposed of by breaking them in the dumpster or recycle containers. Appropriate personal protective equipment (safety glasses, gloves, etc.) must be worn when breaking empty sample containers. All labels must be removed prior to disposal.
  - f. If samples are deemed hazardous, the Laboratory Coordinator will generate an analysis report for the involved samples. The Laboratory Coordinator's signature on this report will be the authorization for disposal of these samples. The specific compounds for which the sample is deemed hazardous should be marked. Any additional information (i.e., known contamination which was not tested for) should also be marked on the analysis report.
  - g. The coldroom technician will turn the samples and the analysis reports over to the hazardous waste technician.
  - h. The hazardous waste technician will store the samples with their report in the hazardous waste storage building until the next hazardous waste pickup.
  - i. During the storage time, the hazardous waste technician will combine all compatible samples to achieve the smallest overall volume.
2. Samples not stored in the coldroom will be handled as follows:
- a. The same criteria for disposal into the waste treatment system or dumpster apply to all samples.
  - b. The responsible Department Manager will give a list of samples to be disposed to the appropriate Laboratory Coordinator.

- c. The Laboratory Coordinator uses the guidelines for sample disposal and his knowledge of the sample to determine if the sample should be classified as hazardous.
- d. The Laboratory Coordinator will generate analysis reports for those samples deemed to be hazardous. The Laboratory Coordinator will mark the specific compounds for which the sample is hazardous on the report. The Laboratory Coordinator's signature will authorize disposal. The Laboratory Coordinator will give the throw-out list and analysis reports back to the Department Manager.
- e. For those samples deemed hazardous, the Department Manager will turn the samples and a signed analysis report over to the coldroom technician for disposal.

## 9.0 CALIBRATION PROCEDURES AND FREQUENCY

Calibration procedures establish the relationship between a calibration standard(s) and the measurement of that standard by an instrument or analytical procedure. At a minimum, calibration is required: (1) when an analytical method is first set up, (2) prior to the analysis of any lot or batch of samples, (3) when the instrument detector has been subject to major maintenance, or (4) when the instrument fails the calibration QC checks.

All analytical instruments are calibrated with each use. A series of standard solutions is prepared from stock standards. These standards are either purchased from various vendors in premixed solutions or prepared directly from the stock compound. The preparation of all standard solutions is documented in a standard preparation logbook. All stock standards are dated when received, opened, and prepared (laboratory). These standards are stored in designated areas and checked for expiration dates. Specific calibration requirements for major classes of analytical procedures are described in the following sections.

### 9.1 STANDARD RECEIPT AND TRACEABILITY

Before any standard is purchased from a supplier, traceability and safety must be considered. This includes a consideration of the standards purity. The purity of the analyte of interest must be known at least to the accuracy requirements for its measurement. The manufacturer ensures this through certification and traceability statements. All laboratory standards must be traceable to a NIST (or EPA equivalent) source. Other chemicals must have a purity specification mentioned on their labels. The safety requirements are checked with the material safety data sheets (MSDS) which are supplied by the manufacturer.

Upon receipt, the standard is cross referenced to its purchase order to confirm that what was received is what was ordered. The chemical is hand delivered (special carrying case if required) to the department manager or analyst. The standard receipt date and initials

are noted on each standard. All standards are stored in designated areas for each department (Table 8-2).

## 9.2 STANDARD SOURCES AND PREPARATION

All standards used in the laboratory must be traceable to a reference source to meet the accuracy requirements as outlined in Section 5.0. The concentrations of the working solutions will depend on the calibration range of each analyte of interest. A standard is a solution of an analyte of interest with verifiable accuracy which is used to evaluate that constituent in a sample.

All new standard preparations are recorded in the appropriate standard preparation logbooks. The information recorded are the standard prepared, the source and concentration of the standard, the standard lot number, date prepared, and initials of the preparer.

The protocols for standard sources and preparation are in Table 9-1. All standards should not exceed the storage (use) life for both the stock and working solutions. Each working solution and stock solution should be labeled with date prepared, initials, concentration used, and expiration date.

Secondary dilutions made from stock standards are also recorded in the standard preparation logbook. The lot number of the stock standard used and the notebook number and/or page number will also be indicated in the logbook for traceability. Table 9-1 lists the frequency of standard preparation and storage of standards by instrument group.

## 9.3 LABORATORY INSTRUMENTS

Calibration criteria will be required for analytical operations. All the laboratory instrumentation is listed in Table 9-2. Each of these instruments will be calibrated in a manner consistent with EPA calibration protocols and/or ESE SOPs. Calibration will be documented in a parameter notebook or the analyst's notebook.

Table 9.1. Standard Sources and Preparation

Instrument Group	Standard Source(s)	How Received	Source Storage	Preparation from Source	Lab Stock Storage	Prep Frequency
ICAP	Various	1,000 ppm soln.	RT	Intermediate and/or Working Stock	RT	> 5ppm Monthly < 5ppm Daily
GFAA					RT	
CVAA					RT	
FLAA					RT	
Autanalyzer (NO <sub>x</sub> , PO <sub>x</sub> , or Phos... CN <sub>x</sub> , TRN, NH <sub>4</sub> , + RH <sub>4</sub> , Phenol)	Various	Neat and/or Solution	RT	Primary Working	RT RT	Monthly Semiannually (CN <sub>x</sub> ) Daily (all others)
IR	Various	Neat (oil)	RT	Intermediate Working	RT RT	Daily Daily
Visible Spectrophotometer UV-VIS (MIRAS, CR <sup>2+</sup> , S. Solgel)	Various	Neat	RT	Combined primary Intermediate Working	RT RT RT	Quarterly Monthly Monthly
TIC Analyzer (TIC, TIC)	Baxter	Neat	RT	Intermediate Working	Refrigerator	Semiannually and Monthly Daily
CO <sub>2</sub> Reaction	Baxter	Neat	RT	Intermediate	RT	Monthly
pH Meter	Baxter	Various Buffers Solutions	RT	Primary	Refrigerator RT	Semiannually Monthly
Specific Ion Meter (Fluoride)	Baxter	Neat	RT	Intermediate	RT	Semiannually
Amperometric Titrator (Acidity)	Baxter	Neat	RT	Intermediate	Refrigerator	Monthly
Turbimeter	Baxter	Formazin Solution	RT	Working	Not stored	Daily
Conductivity Bridge (Specific Conductance)	Baxter	Neat	RT	Primary Working	Refrigerator	Monthly Daily

Table 9-1. Standard Sources and Preparation (Continued, Page 2 of 2)

Instrument Group	Standard Source(s)	How Received	Source Storage	Preparation from Source	Lab Stock Storage	Prep Frequency
GC (non-VOC)	Various	Neat	Freezer	Primary Intermediate	Freezer or Refrigerator Refrigerator	Annually Annually or Semiannually Semiannually
GC (VOC)	Various (Aliphatic Alkyls)	Neat	Freezer	Mixed Primary Working	Refrigerator	Semiannually
LC	Various	Mixed Soda, (Early Gases) Neat	Freezer	Working	Freezer Freezer Freezer	Unmonthly Bimonthly Weekly
GC/MS (non-VOC)	Various (Aliphatic Sulfides)	Mixed Soda	Freezer	Working	Freezer	Annually/Semiannually
GC/MS (VOC)	Various (Aliphatic Sulfides)	Mixed Soda	Freezer	Working	Freezer	Semiannually
Radiochemistry of Compounds	Various	Soda	RT Lead Box	Working	RT Unfilled but monitored	> 50% decrease in activity

Note: RT = Room Temperature  
IR = Infrared

Source: ESLE

Table 9-2. List of Laboratory Instruments

Analysis Type	Number	Instrument
Gas Chromatography/ Mass Spectrometry: Semivolatiles	2	HP 5988 GC/MS/DS capillary direct with HP-7671 Autosampler <sup>III</sup> and HP 5987 GC/MS/DS capillary direct with HP-7671 autosampler <sup>III</sup> ; both instruments share one HP RTE-6/VM HP 1000 computer system for data acquisition and reduction and have one HP 7959 304MB hard drive and one HP 7914 132MB hard drive for a total storage capacity of 463MB.
	2	HP 5970B GC/MS/DS capillary direct with HP 7673 Autosampler; both instruments share an HP RTE-A, HP 1000 computer system for data acquisition and reduction; and have two HP-7959 304MB hard drives for a total data storage capacity of 608MB.
	1	Finnigan INCOSS0 GC/MS/DS capillary direct with HP 7673 Autosampler and HP 5890 gas chromatograph; uses a Data General DG-10 computer with 70MB hard drive; and has an IBM-PC/AT for second terminal.
Volatiles	2	HP 5995 GC/MS/DS <sup>III</sup> using packed column with jet separator interface and HP 5987 GC/MS/DS <sup>III</sup> using packed column with jet separator interface; attached to a Tekmar 2000 liquid sampler (LCS) and Tekmar 2016 sixteen position autosampler (ALS); both instruments share one HP RTE-6/VM, HP 1000 computer system for data acquisition and reduction with HP7920 50MB and HP 7914 132MB hard drives for a total data storage capacity of 314MB.
	1	HP 5970B GC/MS/DS with megabore column (DB-624, 30M x 0.53 mm ID with jet separator interface; attached to Tekmar 2000 LSC and Tekmar 2016 sixteen position ALS; uses a HP RTE-A, HP 1000 computer system for data acquisition and reduction with two HP 7959 304 MB hard drives for a total data storage capacity of 608MB.
	1	HP 5989 Engine GC/MS/DS with capillary direct megabore column, DB-624, 75M x 0.53 mm ID; attached to Tekmar 2000 LSC and Tekmar 2016 sixteen position ALS; uses a HP -425T Apollo workstation, HP-UX UNIX computer system for data acquisition and reduction utilizing the Target 2 and the Envisions software developed by THRU-PUT for HP; the compute system has two HP-6C00 660MB hard drives for a total data storage capacity of 1.3GB.

Table 9-2. List of Laboratory Instruments (Continued, Page 2 of 5)

Analysis Type	Number	Instrument
	1	Finnigan INCOSS500 XL/E GC/MS/DS with megabore column, DB-624, 75M x 0.53 mm ID with jet separator interface and Varian 3400 gas chromatograph; attached to Tekmar 2000 LCS and Tekmar 2016 sixteen position ALS; uses Data General Eclipse MV/1000 computer with 179MB hard drive and a Compaq Desk-Pro 386/20E for second terminal.
Gas Chromatography	10	HP 5890 GC configured for automatic sampling and equipped with dual Electron Capture Detectors. Three of the GCs are attached to a HP 3350 Laboratory Data System (LDS) and 7 to a PE Nelson Data Acquisition System.
	1	HP 5890 GC configured for automatic sampling and equipped with dual Nitrogen-Phosphorus Detector. The GC is attached to a PE Nelson Data Acquisition System.
	1	HP 5890 GC configured for automatic sampling and equipped with Flame Ionization and Flame Photometric Detectors. The GC is attached to a PE Nelson Data Acquisition System.
	1	HP 5890 GC configured for automatic sampling and equipped with Photoionization, Electron Capture, and Flame Ionization Detectors. The GC is attached to a PE Nelson data Acquisition System.
	1	HP 5850 GC configured for automatic sampling and equipped with dual Electron Capture Detectors.
	2	HP 5730 GC configured for automatic sampling and equipped with Electron Capture Detectors. The GCs are attached to a PE Nelson data Acquisition System.
	1	Shimadzu GC-14A configured for automatic sampling and equipped with dual Nitrogen-Phosphorus and Flame Ionization Detector. The GCs are attached to a PE Nelson Data Acquisition System.
	2	Varian 3400 GCs configured for automatic sampling. One GC is equipped with just a Flame-Photometric Detector and the other GC with dual Flame-Photometric and Flame Ionization Detectors.

Table 9-2. List of Laboratory Instruments (Continued, Page 3 of 5)

Analysis Type	Number	Instrument
	4	Tracor 540 GCs equipped with series mounted Photoionization and Electrolytic Conductivity Detectors. Two of the GCs are attached to a Tekmar Purge and Trap LCS 2000 sample concentrator and 16 position Tekmar ALS 2016 automatic sampler. And two of the other GCs are attached to a Tracor Purge and Trap LCS-2 sample concentrator and 10 position Tekmar ALS automatic sampler. The GCs are attached to a PE Nelson Data Acquisition System.
HPLC	1	Shimadzu SCL-6A/SIL-6A (Controller/Injector) Gradient HPLC System with LC-6A pumps (2), ABI/Kratos 520 PCRS (post-column reactor), and Beckman 110B pumps(2); equipped with SPD-551 UV Detector and RF-551 Fluorescence Detector; and attached to a PE Nelson Data Acquisition System.
	1	Shimadzu SCL-6B/SIL-6B (Controller/Injector) Isocratic HPLC System with Beckman 110B pump; equipped with ABI/Kratos 757 UV Detector; and attached to a PE Nelson Data Acquisition System.
	1	Shimadzu SCL-6B/SIL-6B (Controller/Injector) Gradient HPLC System with LC-6A pumps (2); equipped with Kratos 757 UV Detector.
	1	Shimadzu SCL-6A/SIL-6A (Controller/Injector) Isocratic HPLC System with LC-6A pump; equipped with SPD-6AV UV/Visible Detector; and attached to a PE Nelson Data Acquisition System.
	1	Shimadzu SCL-6B/SIL-6B (Controller/Injector) Gradient HPLC System with LC-6A pumps (2); equipped with SPD-6A UV Detector; and attached to a PE Nelson Data Acquisition System.
	1	Hewlett Packard 1090 with ternary gradient solvent delivery, equipped with Diode Array Detector (DAD) and 2 UV/Visible channel outputs.
	1	Shimadzu SCL-6A/SIL-6A (Controller/Injector) Gradient HPLC System with LC-6A pumps (2); equipped with SPD-6AV UV/Visible Detector, RF-555 Fluorescence Detector and Hioko DI constant temperature circulating bath; and attached to a PE Nelson Data Acquisition System.



Table 9-2. List of Laboratory Instruments (Continued, Page 4 of 5)

Analysis Type	Number	Instrument
	1	Shimadzu SCL-10A/SIL-10A (Controller/Injector) Gradient HPLC system with LC-10A pumps (2); equipped with SPD-10A UV Detector; and attached to a PE Nelson Data Acquisition System.
Metals	1	Jarrel-Ash 1100 Inductively Coupled Plasma Emission Simultaneous Spectrophotometer System.
	2	Perkin-Elmer P11 and P40 Inductively Coupled Plasma Emission Sequential Spectrophotometer System.
	2	Perkin-Elmer Model 5100 Atomic Absorption Spectrophotometer System equipped with Flame and Graphite Furnace Model HGA 600 with Zeeman Background Correction.
	1	Perkin-Elmer 5100 Atomic Absorption Spectrophotometer System equipped with Graphite Furnace Model HGA 600 and Zeeman Background Correction.
	1	Perkin-Elmer 3030 Atomic Absorption Spectrophotometer System equipped with Graphite Furnace and Zeeman Background Correction.
	2	Perkin-Elmer Models MHS 20 and 50B Cold Vapor Mercury Analyzer.
	1	Buck Scientific 400 Cold Vapor Mercury Analyzer.
	1	Perkin-Elmer Model 3100 Atomic Absorption Spectrophotometer equipped with Flame, Hydride Generator, and cold Vapor attachment and D <sub>2</sub> background correction.
	1	Perkin-Elmer FIAS-200 automatic flow injection Mercury/Hydride System with amalgamation attachment.
	1	Perkin-Elmer Model 4100 ZL Atomic Absorption Spectrophotometer equipped with Graphite Furnace and Zeeman background correction.

Table 9-2. List of Laboratory Instruments (Continued, Page 5 of 5)

Analysis Type	Number	Instrument	
Inorganics	1	Dorhman DC-190 TOC Analyzer.	
	4	Dionex 4000I (2) and 2000I (2) Ion Chromatographs with autosampler.	
	5	Technicon II (4) and TRACCS 300 (1) Autoanalyzer.	
	3	HACH 16500 COD Reactors.	
	6	Corning 125 (3) pH meters	
	2	Orion 801 and 701 Specific Ion Meters with 6-position electrode switch.	
	1	Perkin-Elmer 1420 Scanning Infrared Spectrophotometer.	
	1	Perkin-Elmer 552 UV-Visible Spectrophotometer.	
	1	Bausch & Lomb Spectronic 20 Visible Spectrophotometer.	
	1	Wallace-Tierman Amperometric Titrator.	
	3	Hach 2100A Turbidimeter	
	1	Hach Ratio Turbidimeter	
	3	Mettler H50 (1), Mettler AE160 (5), and Mettler PC2000 (2) Analytical Balances	
	1	Ohaus Analytical Balance	
	2	Sartorius Analytical Balance	
	Radiochemistry	2	EG&G Berthold LB770-2 10 Channel Simultaneous Low Background Gas Flow Proportional Counter with PC Interface-2.
		1	EG&G Ortec Multichannel Analysis (MCA) System linked with one 20% High Purity Germanium Model GEM20150 Detector, one 35% High Purity Germanium Model GEM 35155 Detector, eight EG&G Ortec Octete PC Model 1000 Detectors Alpha Spectroscopy System, and a Bieron Model P-14-W Sodium Iodide Detector.
1		Beckman LS1501 Liquid Scintillation Detector.	
12		Ludlum Model 312 Alpha Scintillation Detectors with Model 1000 Scalers.	
1		R.J. Harvey OX-600 Biological Oxidizer System.	

The other HPLC equipments we have that can be attached to any of the five HPLC systems, if needed are: RF-535 Fluorescence Detector; Raytest Rannona S-LS Radiochemistry Detector; Beckman 110A pump; Gilson FC-80K fraction collector; Shimadzu FCV-2AH column switching valve; Waters WISP 712 autosampler; Altex 210 manual injector; and Wescan Conductivity Detector.

Specific calibration requirements for major classes of analytical procedures are described in Sections 9.3.1 through 9.3.12. If the calibration requirements of the specified analytical method are more stringent than the procedures described in this LCQAP, the method procedures will be followed.

9.3.1                   GAS CHROMATOGRAPH/HIGH PRESSURE LIQUID  
                          CHROMATOGRAPH (GC-NONVOLATILES/HPLC)  
                          CALIBRATION

Standard Curve Calibration--Initial calibration standard solutions will be prepared by sequential dilution of a single stock standard solution to cover the analytical working range of the method. These may be either composite standards of more than one analyte or single-analyte solutions. The concentrations will be adjusted to take into account the instrumental and method detection limit. A minimum of three initial calibration standard concentrations or the number of standards specified by the method covering the working range and a blank will be prepared and analyzed. The initial calibration standards and the blank will be analyzed in every analytical run. At least one calibration standard at the middle or high range of the curve will be analyzed every 20 samples and repeated at the end of the run. A QC check standard is analyzed every time new calibration standards are prepared and analyzed to verify acceptability of the new calibration standards.

The initial calibration curve will be produced by plotting the standard response for each standard versus the concentration of each standard from the initial calibration run. The concentrations of the standards may be expressed in units of mass injected or in terms of the concentration of the standard solution, if the injection volume is constant for standards and samples. QC evaluation criteria for initial calibration, recalibration, and continuing calibrations are as follows:

1. The initial calibration curve and the subsequent recalibrations possess a minimum of three points and a blank or possess the number of calibration standards specified by the method.
2. The correlation coefficient of the curve is 0.995 or greater.
3. Continuing calibration standards are within 15 percent of the same initial calibration standard for GC (25 percent for NP detector) and within 10 percent of the same initial calibration standard for HPLC.

4. The QC check standard must be within the acceptance range provided by the vendor or within 25 percent of the standard's true if a standard from a different source or lot number is used, and
5. The calibration curve brackets the response for all samples.

Corrective actions taken if these calibration QC criteria are not met are listed in Section 13.0.

The concentration (or amount) of the injected sample will be obtained by entering the response for the sample into the initial calibration curve equation and determining the sample concentration after all appropriate extract and sample dilution factors have been applied.

For Los Alamos project, calibration requirements for organochlorine pesticides and PCBs specified in CLP SOW 12/90 will be followed.

#### 9.3.2 GAS CHROMATOGRAPH (GC-VOLATILES) CALIBRATION

Standard Curve Calibration--Calibration standard solutions will be prepared as needed by sequential dilution of a single stock standard solution (prepared every 2 months) to cover the analytical working range of the method. These may be either composite standards of more than one analyte or single-analyte solutions. The concentrations will be adjusted to take into account the instrumental and method detection limit. A minimum of three calibration standard concentrations, or the number of standards specified by the method covering the working range and a blank, will be prepared and analyzed. The calibration standards and the blank will be analyzed in every analytical run. At least one calibration standard at the middle to high range of the curve will be analyzed every 20 samples and repeated at the end of the run to ensure constant instrument response. A QC check standard is analyzed every time new calibration standards are prepared to verify acceptability of the new calibration standards. Calibration is as described in Section 9.3.1.

### 9.3.3 GAS CHROMATOGRAPH/MASS SPECTROMETER (GC/MS) TUNING AND CALIBRATION

GC/MS Tuning--Daily instrument tuning will be practiced to ensure the instrument is calibrated and in proper working condition. The GC/MS will be tuned daily with decafluorotriphenylphosphine (DFTPP) for semivolatiles analysis and bromofluorobenzene (BFB) for volatiles analysis. The mass intensity specifications for BFB and DFTPP are contained in Table 9-3. For Los Alamos project, the CLP SOW 12/90 mass specifications for BFB and DFTPP will be followed. ESE performs mass calibration in conjunction with the daily instrument tuning.

GC/MS Calibration--Relative response factors for the individual compounds will be determined as follows:

$$RF = \frac{A_C Q_{IS}}{A_{IS} Q_C}$$

- where:
- A = integrated area taken from the extracted ion current profile.
  - Q = quantity of material.
  - C = compound, and
  - IS = internal standard.

Initial calibration, using a minimum of five levels of the compound, will be used to determine the instrument linearity. The average response factor (RF) will be calculated for each compound. The response factors for the System Performance Check Compounds (SPCC) must be  $\geq 0.30$  (0.25 for bromoform) for EPA 8240 and  $\geq 0.05$  for EPA 8270. The percent relative standard deviation (% RSD) will be calculated for each calibration check compound (CCC). The percent RSD of the CCCs in the initial calibration must be  $\leq 30$  percent ( $\leq 35$  percent for EPA Method 625 only).

Table 9-3. Mass Intensity Specifications for DFTPP and BFB

Key Ions	Ion Abundance Criterion
<u>For DFTPP*</u>	
51	30 to 60 percent of mass 198
69	Less than 2 percent of mass 69
70	Less than 2 percent of mass 69
127	40 to 60 percent of mass 198
197	Less than 1 percent of mass 198
198	Base peak, 100-percent relative abundance
199	5 to 9 percent of mass 198
275	10 to 30 percent of mass 198
365	Greater than 1 percent of mass 198
441	Present but less than mass 443
442	Greater than 40 percent of mass 198
443	17 to 23 percent of mass 442
<u>For BFB*</u>	
50	15 to 40 percent of mass 95
75	30 to 60 percent of mass 95
95	Base peak, 100-percent relative abundance
96	5 to 9 percent of mass 95
173	Less than 2 percent of mass 174
174	Greater than 50 percent of mass 95
175	5 to 9 percent of mass 174
176	Greater than 95 percent but less than 101 percent of mass 174
177	5 to 9 percent of mass 176

\*Reference: Test Methods for Evaluating Solid Waste, EPA-SW-846, 3rd Edition, November 1986.

Source: ESE.

A 1-point calibration using a midlevel standard from the initial calibration will be used daily for all subsequent analysis. The RFs of the SPCC for EPA 8240 and 8270 in this continuing calibration standard must meet the minimum response factors specified for the initial calibration previously mentioned. The RFs of the calibration check compounds in this daily calibration standard should be  $\leq 25$  percent ( $\leq 20$  percent for EPA Method 625 only and  $\leq 30$  percent for EPA Method 8270) difference from the average RFs in the initial calibration. All other analytes should have a percent difference of 30 percent. Corrective actions taken if the QC criteria for calibrations are not met are listed in Section 13.0.

The minimum required internal standards (IS) are chlorobenzene-d5, 1,2-dichloroethane-d4, and 1,4-dichlorobenzene-d4, for volatiles and 1,4-dichlorobenzene-d4, naphthalene-d8, acenaphthene-d10, phenanthrene-d10, chrysene-d12, and perylene-d12 for semivolatiles. A retention time and response check will be performed on every internal standard for samples that will be analyzed. The retention time (RT) of the IS in the sample must be  $\leq \pm 30$  seconds from the previous daily calibration. The response area of the IS in the sample should not be  $>$  factor of 2 (-50% to +100%) from the previous calibration.

**9.3.4 GENERAL INORGANIC AND ORGANIC PARAMETERS CALIBRATION**  
Standard Curve Calibration--This section applies to those inorganic and organic analyses procedures [ion chromatography, colorimetric, spectrophotometric, potentiometric, infrared (IR) and ultraviolet (UV) absorption, turbidimetric] that use a standard curve for calibration [except total organic carbon (TOC) and chemical oxygen demand (COD)]. Working standard solutions will be prepared by sequential dilution of a single-stock standard to bracket the analytical working range of the method. Working standard solutions may be either composite standards of more than one analyte or single-analyte solutions. The standard concentrations will be adjusted to take into account the instrument and method, upper and lower limits of linearity, and the instrumental detection limit. A minimum of three standard concentrations, or the number of standards specified by the method, covering the working range and a blank will be prepared and analyzed. The working standards and the blank will be analyzed at the beginning of every analytical run, and at least one midlevel standard, which is the continuing calibration verification

(CCV) standard, will be reanalyzed at minimum intervals of every 20 samples and at the end of the run to check for constant instrument response.

The preparation of calibration standards is verified by the analysis of the ICV solution. The initial calibration verification (ICV) is an independent standard prepared from different stock solutions than those used to prepare the calibration standards. Typically, an EPA or NIST reference is used as the ICV and is prepared according to the supplier's instructions.

The working curve will be produced by plotting the standard response for each standard versus the concentration of each standard from the initial calibration run. QC evaluation criteria for working curves are as follows:

1.  
The working curve possesses a minimum of three points, or the number of standards specified by the method, and a blank;
2. The correlation coefficient of the line is 0.995 or greater;
3.  
The response for the CCV analyzed at minimum intervals of every 20 samples (every 10 samples for cyanide for Los Alamos project) during the run and at the end of the run is within 20 percent of true value (15 percent of true value for cyanide for Los Alamos project);
4.  
The ICV is within 10 percent of the element's true value; and
5.  
The calibration curve brackets the response for all samples.

Corrective action procedures taken if these QC evaluation criteria are not met are provided in Section 13.0. The sample concentration will be obtained by entering the response for the sample into the working curve equation and determining the sample concentration after all appropriate extract and sample dilution factors have been applied.

### 9.3.5 TRACE METALS ANALYSIS CALIBRATION

Atomic Absorption Spectroscopy (AAS) Standard Curve Calibration--Working standard solutions will be prepared to include the analytical working range of the method; these solutions may be either composite standards of more than one metal or single-metal solutions. The standard concentrations will be adjusted to take into account the instrument and method, upper and lower limits of linearity, and the instrumental detection limit. A minimum of three standard concentrations, or the number of standards specified by the method, covering the working range and a blank will be prepared and analyzed. The working standards and the blank will be analyzed at the beginning of every analytical run, and at least one midlevel standard will be analyzed at minimum intervals of every 20 samples (every 10 samples for cyanide for Los Alamos project) during the run and at the end of the run to check for constant instrument response.

The calibration is verified by the analysis of the ICV solution. The ICV is an independent standard prepared from different stock solutions than those used to prepare the calibration standards. Typically an EPA or NIST reference is used as the ICV and is prepared according to the supplier's instructions. For Los Alamos project, the ICV for cyanide analysis should be distilled with the batch of samples analyzed.

The working curve will be produced by plotting the standard response for each standard versus the concentration of each standard from the initial calibration run. QC evaluation criteria for working curves are as follows:

1. The working curve possesses a minimum of three points, or the number of standards specified by the method, and a blank;
2. The correlation coefficient of the line is 0.995 or greater;
3. The response for the midlevel standard analyzed at minimum intervals of every 20 samples (every 10 samples for Los Alamos project) during the run and at the end of the run is within 20 percent of true value (10 percent of true value for GFAA for Los Alamos project);
4. The ICV is within 10 percent (20 percent for CVA.A for Los Alamos project) of the element's true value; and
5. The calibration curve brackets the response for all samples.

Refer to Section 13.0 for the corrective action procedures taken if these QC evaluation criteria for calibration are not met.

The concentration of the sample is obtained by entering the response for the sample into the working curve equation and determining the sample concentration after all appropriate digestate and sample dilution factors have been applied.

Inductively Coupled Argon Plasma (ICAP) Single Point Calibration--This procedure uses a single standard concentration for each element to obtain an instrument response (emission counts) and is analyzed in every analytical run. A second single point, emission counts obtained when aspirating a blank solution (undigested, acidified DI water), is used in conjunction with the standard to calibrate the instrument in concentration units.

The calibration is verified by the analysis of an ICV solution, which is an independent standard prepared from different stock solutions than those used to prepare the calibration standards. The elemental concentrations of the calibration verification solution must be within the calibration range of the instrument and at concentrations other than those used for instrument calibration.

A multi-element interference check solution (ICS) and a method blank (acidified DI water that is carried through the digestion process) are analyzed each day prior to analyzing the samples. For Los Alamos project, the ICS will be analyzed at the beginning and end of each analysis run or a minimum of twice per 8 hour working shift, whichever is more frequent. The ICS is used to verify the correction of spectroscopic interference caused by emissions adjacent to analyte emission lines.

The CCV solution is analyzed at minimum intervals of every 20 samples (10 samples for Los Alamos project) during the run and at the end of the run to document constant instrument response. This solution contains one-half the concentration of each element present in the calibration standards. This solution may be prepared by dilution of an

aliquot of the calibration standard or prepared as a separate solution in a manner analogous to the calibration standard preparation procedure.

QC evaluation criteria for the instrument calibration standard are as follows:

1. A calibration standard and a calibration blank are used.
2. All the values for the ICV are within 10 percent of each element's true value.
3. Values for the ICS are 20 percent of each element's true value.
4. The measured concentrations of the elements in the CCV solution, for which calibration was performed, are within 10 percent of their respective true values.

Corrective action procedures if these QC evaluation criteria are not met are provided in Section 13.0.

### 9.3.6 GRAVIMETRIC METHODS CALIBRATION

Two general types of analytical balances are used at ESE: (1) the more sensitive microanalytical balance, and (2) the top-loading balance. The calibration of the microanalytical balances is verified daily by weighing the following Class S and NIST-certified weights (in grams (g)):

<u>Weight (g)</u>	<u>Tolerance Limits</u>
0.2	$\pm 0.0005$
1.0	$\pm 0.0005$
3.0	$\pm 0.0005$
5.0	$\pm 0.0005$

The calibration of the top loading balances are verified daily by weighing the following Class S and NIST-certified weights:

<u>Weight (g)</u>	<u>Tolerance Limits</u>
5	$\pm 0.02$
20	$\pm 0.05$
50	$\pm 0.05$

The results are recorded in the instrument logbook. If these criteria are not met, the weight may be reweighed. If the criteria are not met for the second weighing, the balance is taken out of service and repaired. The Class S weights are sent to the manufacturer yearly for calibration and recertification. Two sets of Class S weights are available in-house.

Qualified service personnel calibrate the analytical balances semiannually. The semiannual calibration is documented by a tag on the instrument. A set of NIST-certified weights is used to check the calibration daily. Results are recorded in the instrument notebook.

#### 9.3.7 TITRIMETRIC METHODS CALIBRATION

In all cases, standard reference materials are used to calibrate the titrant and back titrant. Preparation of these materials is described in Standard Methods or other methods manuals. Known solutions of the parameter to be analyzed will be prepared and analyzed to verify titrant standardization and the analyst's ability to discern the endpoint.

#### 9.3.8 TOC CALIBRATION

The Dohrman TOC analyzer is calibrated with a standard reference material using a single-point calibration. The standard is analyzed before beginning every analytical run. The linearity of the calibration is verified with a low-level and high-level standard to bracket the sample concentration. The linearity checks must be within 5 percent. The continuing calibration verification standard (using mid- to high-level standard) is analyzed every 10 samples and at the end of the run, and the response must be within  $\pm 15$  percent of true value.

#### 9.3.9 COD CALIBRATION

A reference material will be used to verify the 0- and 500-mg/L reading to the standard curve developed by Hach Chemical Company for COD on a spectrophotometer using the prepared sample vials. The 500-milligrams-per-liter (mg/L) standard must be within 5 percent.

#### 9.3.10 BOD CALIBRATION

The oxygen probe is calibrated daily according to the manufacturer's air calibration procedure. The temperature of the incubator used for the BOD analysis will be read and recorded twice daily when in use.

#### 9.3.11 TOTAL ORGANIC HALIDES (TOX) CALIBRATION

The TOX analyzer is calibrated with a standard reference material using a 3-point calibration. The linearity of the calibration is verified with a low-level and high-level standard to bracket the sample concentration. The linearity checks must be within 5 percent. The continuing calibration verification standard (using mid- to high-level standard) is analyzed every 10 samples, and the response must be within  $\pm 15$  percent of true value.

#### 9.3.12 RADIOCHEMISTRY CALIBRATION

In compliance with the State of Florida DHRS Radioactive Materials licensing regulations, control charts for efficiencies and backgrounds are kept for all instruments used in radiochemical counting. All standards used in the calibrations and QC spiking are either from NIST or EPA. Count rates are calculated using computer software.

Alpha/Beta Proportional Counter--The 10-chamber, low-background alpha/beta proportional counting system is calibrated for counting efficiencies of all radionuclides on an annual basis with Am-241 and Cs-137 standards. The calibration efficiencies are verified daily by counting control check samples. Results of the check samples must be within historical control limits. The alpha/beta self-absorption calibration curve for each counting chamber and the beta spillover curves are determined annually. The voltage plateau is generated annually and verified whenever the gas is changed by counting control check samples. The voltage of the check sample must be within  $\pm \geq 50V$  from the operating voltage. Background is also checked prior to each analytical run, and the count rate must be within historical control limits. Historical limits for the control check standards and backgrounds are generated quarterly.

Liquid Scintillation Counter--Calibration efficiencies of the Beckman Liquid Scintillation Counting System for specific radionuclides are performed annually. A control check sample is analyzed prior to each instrument use to verify the calibration efficiency, and count rates must be within historical control limits generated quarterly. The instrument is also calibrated prior to each run with applicable standards (tritium, C-14, Pb-210, and Ra-226) provided by the manufacturer. The H Number Quench Efficiency Correction Curve generated annually is derived from a set of quenched standards and applied to the data to account for counting efficiencies. Background measurements are performed prior to each instrument use.

Lucas Cell Readers for Ra-226 Counting--Each Lucas Cell and the 12 matching cell readers are calibrated with known Ra-226 standard annually. The performance check standard is analyzed prior to each instrument use, and count rates must be within historical control limits generated quarterly. A background check is also performed prior to each instrument use, and the reading must be  $\leq$  1 cpm.

Gamma Spectroscopy--The efficiency calibration for all geometries are performed on an annual basis. Efficiency checks are performed daily using four peaks and documented in the gamma spectroscopy daily control logbook. Energy calibration is done monthly and is checked daily using a standard source with four peaks. A resolution check for the four peaks is performed daily. Performance standards are counted prior to each instrument use, and count rates must be within historical control limits generated quarterly. Background measurements are performed weekly or prior to each instrument use.

Alpha Spectrometers-- Alpha spectrometers are calibrated annually for efficiency with known electroplated standard sources. The efficiency is verified monthly by analyzing a control check sample. The count rates of the control check sample must be within historical control limits generated quarterly. Energy calibrations of the alpha spectrometers are performed quarterly and the calibrations are verified weekly using the control check sample. The control check sample must be within historical control limits. Detectors are checked for performance weekly by counting standard sources, and count rates must be within historical control limits generated quarterly. Background

measurements are performed weekly, and the count rate must be  $\leq 0.02$  cpm in any channel.

#### 9.3.12.1 TRACER AND CARRIER RECOVERY ACCEPTANCE CRITERIA

All alpha activity measurements by alpha spectrometry require the use of another isotope of the same element as a tracer. A known activity of the tracer is added to the sample at the beginning of the analysis and subsequently measured by alpha spectrometry. Recovery of this tracer should be at least 50%. Samples with tracer recoveries less than 50% require reanalysis.

Analysis of other radionuclides require the addition of other elements as chemical carriers. The following is a list of the common chemical carriers used for the listed isotopes and their required minimum percent recoveries:

##### Radium-228

Yttrium Carrier	50% Recovery
Barium Carrier	50% Recovery

##### Strontium-89/90

Yttrium Carrier	50% Recovery
Barium Carrier	50% Recovery

##### Lead-210

Lead Carrier	50% Recovery
Bismuth Carrier	50% Recovery

##### Uranium-234, 235/236, 238

Uranium-232 Carrier	50% Recovery
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Thorium-227, 228, 230, 232

Thorium-234 Carrier 50% Recovery

Plutonium-238, 239/240, 241

Plutonium-242 Carrier 50% Recovery

Polonium-210

Polonium-209 50% Recovery

Americium-241

Americium-243 Carrier 50% Recovery

Samples with carrier recoveries less than the above minimum recoveries require reanalysis.

#### 9.3.13 pH CALIBRATION

Calibrate the pH meter with three buffer solutions at pH 4, 7, and 10 prior to use. Set the pH meter temperature selector to ambient temperature. Place the probe on the pH 7 buffer and adjust the calibration switch until it reads 7.00. Repeat the procedure with the pH 4 buffer solution. Read the pH of the pH 10 buffer solution. It should be  $10 \pm 0.05$ ; if not, check the pH probe and internal solution and repeat the calibration procedure.

#### 9.3.14 SPECIFIC CONDUCTIVITY CALIBRATION

Calibrate the instrument with 0.01 M and 0.10 M KCL solutions. The conductivity reading of the 0.01 M KCL must be 1.413 umhos  $\pm 15\%$  and the 0.10 M KCL 12.900  $\pm 15\%$ . If the calibration standards are outside the acceptance criteria, prepare new standards and recalibrate the instrument.

#### 9.3.15 PENSKY-MARTENS CLOSE-CUP TESTER CALIBRATION

Determine the ignitability of the p-xylene standard prior to use of the Pensky-Martens Close-Cup Tester. The standard should ignite at  $27.2 \pm 1^\circ\text{C}$ . If not, check the

condition and operation of the apparatus, especially the tightness of the lid, the action of the shutter, and the position of the test flame. After adjustment, repeat the test with the p-xylene standard. Read and record the barometric pressure at the time of analysis.

#### 9.3.16 DISSOLVED OXYGEN CALIBRATION

The dissolved oxygen probe should be calibrated daily or prior to use in saturated air by moving the calibration knob such that the reading is at the appropriate saturation value indicated on the instrument. Read and record the temperature at the time of reading.

#### 9.4 STANDARDIZATION OF TITRATION SOLUTIONS

All titrants used in the laboratory are standardized against a primary standard. This ensures that the normality of the standard being used is at the correct level. Table 9-6 lists the solutions that require standardization, the standards used, and the frequency of standardization.

Table 9-4. Standardization of Titrating Solutions

Solutions Req.	Primary Standard Source	Frequency of Standardization
Chloride: Silver nitrate	Sodium chloride	Every run
Alkalinity: Sulfuric acid	Sodium carbonate	Every run
Sulfite: Potassium iodide-iodate	Sulfamic acid	Every run
Hardness: EDTA	Calcium carbonate	Every run

Source: ESE.

## 10.0 PREVENTIVE MAINTENANCE

To minimize the occurrence of instrument failure and other system malfunctions, a preventive maintenance program for laboratory instruments is implemented. Routine maintenance is performed as needed depending on how often the instrument is used. There are some parts of the instrument that will wear out faster and therefore will require replacement more frequently than the others. These wearable or expendable parts are kept in supply and evaluated during analysis. The major instrumentation in the laboratory is covered by the manufacturer's service contracts or agreements.

### 10.1 DOCUMENTATION

All maintenance performed on the instruments are documented in each instrument's maintenance logbook which is kept with the instrument. The date, initials of the analyst performing the maintenance, and the type of maintenance performed are recorded in this maintenance logbook. Receipts from the routine maintenance performed by the manufacturer's representative are kept in folders and filed in the department's file cabinets. Preventive maintenance for each major piece of laboratory equipment is listed in Table 10-1.

### 10.2 CONTINGENCY PLAN

In the event of instrument failure, every effort will be made to analyze samples within holding times by alternate means. If the redundancy in equivalent instrumentation is insufficient to handle the affected samples, efforts will be made to secure the same or equivalent analyses by a Navy, HAZWRAP, USACE or FDER-approved laboratory, when required. The Project Manager will be advised of any required changes in methodology or location; the Project Manager should then notify the state or government agency or the client.

Table 10-1. Preventive Maintenance

Instrument	Activity	Frequency
Gas Chromatographs	Change septums	Weekly or as needed
	Check carrier gas	Daily
	Change carrier gas	As needed (when pressure falls below 100 psi)
	Cut off edge of a capillary column	As needed
	Replace oxygen traps used in the gas lines	Annually or as needed
	Clean ECD	Annually or as needed
	Replenish Electrolytic Conductivity Detector	Monthly or as needed
	Clean detectors	Annually or as needed
	Check system for gas leaks	At each column change
	High Performance Liquid Chromatographs	Replace piston seals
Replace or rebuild the the check valves		As needed (when performance of the instrument decreases)
Clean detector flow cell		As needed
Check pumps		As needed
Replace guard column frits		As needed (when the HPLC system pressure increases)
Clean detectors		Annually or as needed
Gas Chromatograph/Mass Spectrometer	Clean source and system	As needed
	Cut off ends of capillary columns	As needed
	Change columns	As needed
	Change injection point lines	Monthly or as needed
	Routine maintenance performed by the manufacturer	Annually
Atomic Absorption Spectrophotometers (Furnace and Cold Vapor)	Clean furnace windows	Daily
	Check plumbing connections	Daily
	Change graphite tubes	Daily or as needed
	Clean sample cells	Daily
	Check gases	Daily
	Check optics	Annually (on contract)
	Change graphite contact rings	As needed
Inductively Coupled Plasma (ICAP)	Routine maintenance performed by the manufacturer	Annually (on contract)
	Clean the torch and nebulizer	Every six months or as needed
Check tubing	Daily	

Table 10-1. Preventive Maintenance (Continued, Page 2 of 3)

Instrument	Activity	Frequency
Autoanalyzers	Clean tubing	As needed
	Check tubings	Daily
	Check optics	Daily
Colorimeter/ Turbidimeters	Clean optics	As needed
	Replace the lamp	As needed (when darkening is evident)
	Check optics	Daily
Spectrophotometer	Check light source	As needed
	Routine maintenance performed by the manufacturer	Quarterly (on contract)
TOX Analyzer	Clean electrodes	Daily
	Replace all solutions	Daily
	Clean absorber module and the furnace unit	Every six months or as needed
	Clean sampler boat	Monthly
	Check gases and tubing	Daily
TOC Analyzer	Check gases and tubing	Daily
	Change pump tubes	Prior to each use
	Flush Digestion tubes	After each use
Ionanalyzers/Conductivity	Check probe	Daily
	Change probe solution	As needed
Ion Chromatograph	Routine maintenance performed by the manufacturer	Every six months (on contract)
	Check system for leaks	Daily prior to each run
	Check line pressure	Daily prior to each use
	Clean conductivity cells	Every six months
	Clean injection loops	Every six months
	Change columns	As needed
	Replace tubings in the sample path	Every six months
	Clean the instrument	Prior to each use
DO Meter and Probe	Check to make sure that the mechanical zero is set properly	Prior to each use
	Check DO probe membrane	Prior to each use

Table 10-1. Preventive Maintenance (Continued, Page 3 of 3)

Instrument	Activity	Frequency
DO Meter and Probe (cont)	Replace membrane	As needed (when tears, wrinkles, or bubbles are observed)
	Replace probe	As needed
Analytical Balances	Clean the balance	Daily
	Check alignment and balance	Daily
	Routine maintenance and calibration performed by the manufacturer	Semiannually
Radiochemistry: Alpha/Beta Proportional Counter	Check gas flow	Daily
	Check counting chambers	Monthly or as needed
Liquid Scintillation Counter	Check counting system	Prior to each use
Alpha Spectrometer	Clean detectors	As needed
	Clean sample chambers	As needed
	Check vacuum	Daily
	Check voltage	Daily
Radon Flask Counters	Clean the face of the photomultiplier tube	Daily
	Check microswitch	Daily
Gamma Spectroscopy	Refill liquid nitrogen in the dewar for the Ge(Li) detector	Weekly
	Check all cabling to the gamma detectors	Monthly
Biological Oxidizer	Clean lidel	Prior to each use
	Clean sample boats	Prior to each use
Ovens: TS, TSS, TDS	Check temperature	Prior to use
	Calibrate thermometer	Annually
Refrigerators/Freezers	Check temperature	Daily
	Calibrate thermometer	Annually
BOD Incubator	Check temperature	Prior to use
	Calibrate thermometer	Annually

Note: TDS = total dissolved solids, TS = total solids, TSS = total suspended solids.

## 11.0 QC CHECKS, ROUTINES TO ASSESS PRECISION AND ACCURACY, AND CALCULATION OF METHOD DETECTION LIMITS

### 11.1 INTERNAL QC CHECKS

Analytical QC procedures are those steps taken by the laboratory in day-to-day activities to achieve the desired accuracy, precision, reliability, and comparability of analytical data. Each Laboratory Department Manager and coordinator is responsible for performing the analysis in accordance with the defined quality control practices outlined in this LCQAP.

For all analyses performed by ESE, the QC checks described in this section are mandatory unless alternate procedures are given in the specific project QA Plan or otherwise agreed upon by the Laboratory Coordinator and the Project QA Officer. Table 11-1 summarizes minimum QC sample requirements. If method QC requirements are more stringent than those listed in Table 11-1, the method requirements will be followed. Sections 3.0 and 5.0 contain QC evaluation criteria for laboratory methods and calibrations. Section 11.4 describes precision and accuracy calculations used to control samples. Laboratory Department Managers are responsible for reviewing QC criteria for each method performed by their department. Permanent changes to the acceptance criteria must be approved by the Laboratory Department and Division Managers and will be incorporated into this document in accordance with Section 3.4, Document Control. Project-specific revisions may be documented in the specific project QA Plan (DER Form 17-160.900 for FDER projects only).

For QC purposes, the number of samples extracted and/or prepared for instrumental analysis as one group in one 24-hour period will constitute a batch. The number and type of QC samples specified in Section 11.0 will apply to this sample batch. For example, a group of samples that is extracted on the same day and (if required) undergoes

Table 11.1. Minimum QC Sample Requirements\*

Analysis	Standard Matrix (QC Check Standard)		Sample Matrix**					Filter Blank (as required)
	Blank	Spike**	Spike	Replicate Spike	Sample Replicate	Surrogate Spike		
<b>INORGANIC</b>								
*All analyses except lead	\$3	\$5	\$5	\$5****	..	..	\$3	
*pH, residue, specific conductivity, turbidity, dissolved oxygen	\$3	..	..	..	\$2	..	..	
*Radiochemistry only	\$3	\$2	\$3	\$3****	100%***	..	\$2	
*TECP	\$3	\$5	..	..	..	..	\$3	
<b>ORGANIC</b>								
*All analyses	\$3	\$5	\$3****	\$3****	..	100%***	\$3	
*TECP	\$3	\$5	..	..	..	100%***	\$3	

Notes: \*\* = not applicable for this analysis.

\*Standard Matrix Spike (QC Check Standard) is a spike into a blank matrix which is carried through sample preparation, sample digestion or extraction to sample analysis. The blank matrix is a reagent blank for aqueous samples and a standard soil for solid matrix. If available, if standard soil is not available, spiking is done on a reagent blank. This spike is also called a QC Check Standard because the standards used to prepare the spiking solution are from a different source than those used for the calibration standards.

\*\*Actual number rounded up to nearest whole number, i.e., \$2 = 10% for 1-30 samples; 20% for 21-40, etc.

\*\*\*Surrogate(s) will only be spiked into all environmental and QC samples if specified by the method.

\*\*\*\*For Los Alamos project, sample matrix spike and sample matrix spike duplicate for organic analysis are not required, however, they will be analyzed if requested by the client.

Source: ES&E.

concentration and cleanup procedures on subsequent days would be considered one sample lot for QC purposes.

For analyses where no sample extraction or preparation is required, the number of samples that can be analyzed as one set during a 24-hour period will determine the number of samples per sample lot for QC purposes.

When required by the specific project QA Plan, the Project QA Coordinator may insert into a sample lot either a spiked sample or a duplicate of a previously analyzed sample for QC purposes. The Project QA Coordinator will monitor the results of this sample to ensure that the analysis meets QA criteria for the project.

Blind QC check samples are analyzed by the laboratory semiannually to evaluate the laboratory's overall system. If the blind QC check sample data are not acceptable, results will be reported in the QA report to FDER.

Spikes will be placed into sample matrices for all analyses except pH, residues, specific conductivity, and turbidity. Samples will be split into duplicates, spiked, and analyzed. The relative-percent difference between the spike and the replicate spike will be used to assess analytical precision. Selection of the sample to be split and spiked may be made by the client or by the laboratory.

Control spikes (standard matrix spikes or QC check standards) will be placed into standard matrices for all analyses except pH, residues, specific conductivity, and turbidity. This spike will be used to control the method and verify the calibration standards, if an ICV is not analyzed. A sample replicate will be prepared and analyzed for pH, residues, specific conductivity, and turbidity. The relative-percent difference between the sample and the replicate will be used to assess analytical precision.

It is ESE's policy to control sample analyses on those QC criteria that are actually under the control of the technicians and analysts performing the analytical procedure. Therefore, emphasis is placed on calibration, method blanks, and QC check standard (standard matrix spike) results. When these are within criteria, acceptable method performance is documented. Sample matrix spikes will be reported and evaluated for precision and accuracy but not necessarily used for method control. A sample matrix spike that has recoveries outside of criteria limits will be evaluated against other available QC data within a batch to determine if the method is in control and if sample flagging is warranted. Failure of a sample matrix spike to achieve the acceptance criteria when a QC check sample in the same batch has acceptable recoveries often documents only that the method employed is not applicable to that particular matrix, not that the method is out of control.

In the following subsections, those criteria marked with an asterisk (\*) will be used to control the sample analysis. Precision and spike recovery checks are discussed in further detail in Section 11.4. In addition to the QC samples specified in the following subsections, field QC blanks must be prepared and analyzed as described in Section 11.1.

#### 11.1.1 GC/MS MINIMUM QC

For GC/MS analyses, the following minimum QC checks will apply, except for CLP SOW:

1. All samples spiked with surrogates.
2. At least 5 percent spikes in sample matrix (MS) with selected analytes and surrogates will be analyzed (analyzed only if requested for Los Alamos project).
3. At least 5 percent duplicate spikes in sample matrix (MSD) with selected analytes and surrogates will be analyzed (analyzed only if requested for Los Alamos project).
4. At least 5 percent QC check spikes in blank matrix with selected analytes and surrogates will be analyzed.

5. At least 5 percent method blanks spiked with surrogates will be analyzed.
6. One calibration standard will be run daily. Response factors must be within 25 percent (20 percent for EPA 625) of initial calibration response factors for selected calibration check compounds. Response factors of the SPCC must be  $\geq 0.05$  for EPA 8270 and  $\geq 0.30$  for EPA 8240 (0.25 for bromoform)
7. Instrument tuning protocols will be performed and be within criteria prior to analysis.
8. Continuing calibration standard will be analyzed at a frequency of 5 percent.

#### 11.1.2 GC AND HPLC MINIMUM QC

For GC-nonvolatiles, GC-volatiles, and HPLC analyses the following minimum requirements will apply, except for CLP SOW:

1. All samples spiked with surrogate, if specified by the method.
2. At least 5 percent spikes in sample matrix (MS) with selected analytes and surrogate(s) (if applicable) will be analyzed (analyzed only if requested for Los Alamos project).
3. At least 5 percent duplicate spikes in sample matrix (MSD) with selected analytes and surrogate(s) (if applicable) will be analyzed (analyzed only if requested for Los Alamos project).
4. At least 5 percent QC check spikes in blank matrix with selected analytes and surrogate (if applicable) will be analyzed.
5. At least 5 percent method blanks spiked with surrogates (if applicable) will be analyzed.
6. At least three standards or the number of standards specified by the method will be analyzed as a standard curve.
7. Correlation coefficient of the standard curve will be equal to or greater than 0.995.
8. Samples will be within concentration range of the standards.
9. Midlevel calibration standards will be repeated at minimum intervals of every 20 samples and at the end of a run, and response of the control analytes must be within 15 percent of initial response for GC (25 percent for NP detector) and 10 percent of initial response for HPLC.

10. Detection limits for each parameter will be determined and checked to ensure they meet limits specified for the field group.

### 11.1.3 TRACE METALS--ATOMIC ABSORPTION AND ICAP SPECTROSCOPY MINIMUM QC

For each batch of samples analyzed by AAS or ICAP, the following QC checks will apply, except for CLP SOW:

1. At least 5 percent spikes in sample matrix (MS) with selected elements will be analyzed.
2. At least 5 percent duplicate spikes in sample matrix (MSD) with selected elements will be analyzed (analyzed only if requested for Los Alamos project).
3. At least 5 percent QC check spikes in blank matrix with selected elements will be analyzed.
4. At least 5 percent method blanks will be analyzed.
5. At least three standards or the number of standards specified by the method will be analyzed as a standard curve.
6. Correlation coefficient of the standard curve will be equal to or greater than 0.995.
7. Samples will be within concentration range of the standards (or of the ICAP instrument).
8. Midlevel calibration standards will be repeated at minimum intervals of every 20 samples (10 samples for Los Alamos project) and at the end of a run, and response of the control elements must be within 20 percent (10 percent for ICAP and GFAA) of true value.
9. At least 5 percent filter blanks will be analyzed with all filtered samples.
10. Detection limits for each element will be determined and checked to ensure they meet limits specified for the field group.

#### 11.1.4 MISCELLANEOUS METHODS MINIMUM QC

For each batch of samples analyzed by ion chromatographic, colorimetric, spectrophotometric, turbidimetric, IR, UV absorption, radiochemical, and titrimetric methods (except for pH, residues, specific conductivity, turbidity, and DO), the following QC checks will apply:

1. At least 5 percent QC check spikes in standard matrix will be analyzed.
2. At least 5 percent sample matrix spikes (MS) will be analyzed.
3. At least 5 percent duplicate control spikes in sample matrix (MSD) will be analyzed (analyzed only if requested for Los Alamos project).
4. For radiochemistry methods, at least 10 percent sample replicates will be analyzed for drinking water samples and Los Alamos project.
5. At least 5 percent method blanks will be analyzed.
6. At least three standards or the number of standards specified by the method will be analyzed as a standard curve.
7. Correlation coefficient of the standard curve will be equal to or greater than 0.995.
8. For radiochemistry methods, performance check standards will be analyzed and background checks performed at the required frequency.
9. Samples will be within concentration range of the standards.
10. Midlevel calibration standards will be repeated at minimum intervals of every 20 samples (10 samples for cyanide for Los Alamos project) and at the end of a run, and responses must be within 20 percent (10 percent for cyanide for Los Alamos project) of true value.
11. At least 5 percent filter blanks will be analyzed with all filtered samples.
12. Detection limits for analytes will be determined and checked to ensure they meet limits specified for the field group.

For each batch of samples analyzed for pH, residues, specific conductivity, turbidity, and DO, the following QC checks will apply:

1. At least 5 percent sample replicates will be analyzed.

2. At least 5 percent method blanks will be analyzed.
3. At least 5 percent filter blanks will be analyzed with all filtered samples.
4. Detection limits for analytes will be determined and checked to ensure they meet limits specified for the field group.
5. Continuing calibration standards will be analyzed at a frequency of 5 percent.

## 11.2 ROUTINE METHODS USED TO ASSESS PRECISION AND ACCURACY

### 11.2.1 PRECISION

Precision is a measure of agreement among measurements performed using the same test procedure. Precision will be assessed for applicable parameters by calculating the RPD of two duplicate spike samples as follows:

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

where:  $R_1$  and  $R_2$  = concentration of Replicate Spikes 1 and 2, respectively.

This calculated RPD value is compared to the criteria specified in this LCQAP. The procedures used to determine the precision targets are listed in Table 11-2.

### 11.2.2 ACCURACY

Accuracy is the degree of agreement between a sample's target value (known concentration) and the actual measured value. Accuracy for this project is measured by calculating the percent recovery (R) of known levels of spike compounds into appropriate sample matrices. Percent recovery is calculated as follows:

$$R = \frac{100 \times [(Spike Sample Conc. \times Sample + Spike Vol.) - (Sample Vol.) (Sample Conc.)]}{(Spike Conc. \times Spike Volume)}$$

The following equation is an example of how this would be calculated:

1 mL of spike with concentration of 100 ppb  
10 mL of sample with concentration of 10 ppb  
spiked sample concentration of 20 ppb

$$= 100 \times \frac{(20)(11) - (10)(10)}{(1)(10)} = 100 \times \frac{120}{100} = 120 \text{ percent}$$

Each calculated R value is compared to the accuracy criteria listed in Section 5.0. The accuracy ranges provided in Section 5.0 are based on the mean accuracy measured or expected (based on EPA data) for each parameter plus or minus three standard deviations of the mean. The procedures used to determine the accuracy targets are listed in Table 11-2. If RPD or R values for standard spikes or sample matrix spikes within a batch do not meet acceptance criteria for QC check samples as specified in Section 5.0, results reported for samples in this batch may require flagging and/or re-analysis. The Laboratory QC Manager or designee will be notified and the necessary corrective action implemented.

### 11.2.3 CONTROL CHARTS OF ACCURACY

Control charts will be maintained for standard laboratory spike samples for FDER, Navy, and other specified programs. Initial control charts are prepared using historical ESE data, or are derived from published EPA method data if sufficient inhouse data are unavailable. Control chart limits are updated yearly or more often as needed using historical data.

Control charts are graphical "pictures" that demonstrate statistical control, monitor trends in a measurement process, and diagnose a measurement problem.

The formulas used to establish and maintain control charts for standard laboratory spike QC samples are as follows:

$$USL_{\bar{x}} = \bar{X} + 3SD$$

$$UWL_{\bar{x}} = \bar{X} + 2SD$$

$$LWL_{\bar{x}} = \bar{X} - 2SD$$

$$LCL_{\bar{x}} = \bar{\bar{X}} - 3SD$$

where:  $\bar{X}$  = mean of the recoveries of the laboratory spikes.  
SD = Standard deviation of the mean.  
UCL = Upper control limit.  
UWL = Upper warning limit.  
LWL = Lower warning limit, and  
LCL = Lower control limit.

All recoveries will be plotted on the appropriate matrix-specific control charts.

Table 11-1. Methods Used to Generate Precision and Accuracy Targets

Method	Purpose	Concentration Level	Method References
Sample Duplicate	Precision	NA	STMD2216, ASTM D 32974, 110.2, 1110, SNI 2350, 360.1, 150.1, 9040, 9045, 160.1, 160.1, 160.3, 900.0, 903.1, 904, 905, 908
Standard Matrix Spike (QC Check Standard)	Accuracy	Low Level	7481, 270.2, 7740, 286.2, 7911, 8240
		Mid Level	200.7, 202, 204.2, 206.2, 208.2, 210.2, 213.2, 218.1, 219.2, 220.1, 239.2, 243.2, 245.1, 249.2, 258.1, 272.2, 273.1, 279.2, 283.2, 286.2, 6010, 7020, 7041, 7060, 7091, 7131, 7191, 7201, 7200, 7210, 7421, 7460, 7470, 7471, 7480, 7610, 7770, 7841, 310.0, HACH 8000, 335.3, 9010, 300, 325.3, 353.2, 9200, 350.1, 351.2, CE-81-1 P 3-201, 365.1, 370.1, 375.4, 376.2, 9030, 305.1, 405.1, 9060, 330.1, 7196, 340.2, 130.2, 413.1, 9071, 9073, 420.1, 9066, 425.1, 413.1, 9071, 9073, 9020, 900.0, 9310, 9315, 904.0, 905.0, 908.0, 501.2, 504, 505, 507, 515.1, 524.2, 531.1, 601, 8010, 602, 8020, 604, 8040, 605, 606, 8060, 608, 617, 8080, 610, 8310, 615, 8150, 622, 614, 8140, 624, 8240, 8260, 625, 8270, 632, 612, 8120, 619, 633, 645, 8350, UW32, LW12, 547, 630.1 (Mid)
		High Level	903.0, 9320
Sample Matrix Spike	Accuracy	Low Level	7481, 270.2, 7740, 286.2, 7911, 8240
		Mid Level	200.7, 202, 204.2, 206.2, 208.2, 210.2, 213.2, 218.1, 219.2, 220.1, 239.2, 243.2, 245.1, 249.2, 258.1, 272.2, 273.1, 279.2, 283.2, 286.2, 6010, 7020, 7041, 7060, 7091, 7131, 7191, 7201, 7200, 7210, 7421, 7460, 7470, 7471, 7480, 7610, 7770, 7841, 310.0, HACH 8000, 335.3, 9010, 300, 325.3, 353.2, 9200, 350.1, 351.2, CE-81-1 P 3-201, 365.1, 370.1, 375.4, 376.2, 9030, 305.1, 405.1, 9060, 330.1, 7196, 340.2,

Table 11-1. Methods Used to Generate Precision and Accuracy Targets (Continued, Page 2 of 2)

Method	Purpose	Concentration Level	Method References
Sample Matrix Spike Duplicates	Precision		130.2, 413.1, 9071, 9073, 420.1, 9066, 425.1, 418.1, 9071, 9073, 9020, 900.0, 9310, 9315, 904.0, 905.0, 908.0, 501.2, 504, 505, 507, 515.1, 524.2, 531.1, 601, 8010, 602, 8020, 604, 8040, 605, 606, 8060, 608, 617, 8080, 610, 8310, 615, 8150, 622, 614, 8140, 624, 8240, 8260, 625, 8270, 632, 612, 8120, 619, 633, 645, 8330, UW32, LW12, 547, 630.1 (Mod)
		High Level	903.0, 9320
		Low Level	7481, 270.2, 7740, 286.2, 7911, 8240
		Mid Level	200.7, 202, 204.2, 206.2, 208.2, 210.2, 213.2, 218.1, 219.2, 220.1, 239.2, 243.2, 245.1, 249.2, 258.1, 272.2, 273.1, 279.2, 283.2, 286.2, 6010, 7020, 7041, 7060, 7091, 7131, 7191, 7301, 7200, 7210, 7421, 7460, 7470, 7471, 7480, 7610, 7770, 7841, 310.0, HACH 8000, 335.3, 9010, 300, 325.3, 353.2, 9200, 350.1, 351.2, CE-81-1 P 3-201, 365.1, 370.1, 375.4, 376.2, 9030, 305.1, 405.1, 9060, 330.1, 7196, 340.2, 130.2, 413.1, 9071, 9073, 420.1, 9066, 425.1, 418.1, 9071, 9073, 9020, 900.0, 9310, 9315, 904.0, 905.0, 908.0, 501.2, 504, 505, 507, 515.1, 524.2, 531.1, 601, 8010, 602, 8020, 604, 8040, 605, 606, 8060, 608, 617, 8080, 610, 8310, 615, 8150, 622, 614, 8140, 624, 8240, 8260, 625, 8270, 632, 612, 8120, 619, 633, 645, 8330, UW32, LW12, 547, 630.1 (Mod)
High Level	903.0, 9320		

Notes: Low Level = Concentration from the reporting limit to 5 times the detection limit.  
 Mid Level = The mean level between the reporting limit and the upper end of the linear range.  
 High Level = Concentration at the upper end of the linear range.

An out-of-control situation for accuracy control charts may be indicated by the following:

1. Any one point plots outside the control limits.
2. Any eight consecutive points plot on the same side of the mean.
3. Any six consecutive points trend in the same direction.
4. A cyclical pattern is evident.
5. Any three consecutive points plot within the control limits but outside the warning limits.

The occurrence of any of these events will be investigated; corrective actions will be taken as required to return the system to a state of statistical control. All corrective actions will be documented.

### 11.3 METHOD DETECTION LIMITS AND PRACTICAL QUANTITATION LIMITS

#### 11.3.1 METHOD DETECTION LIMITS (MDLS)

The detection limit of the method is the lowest sample concentration which can be reliably recovered and measured in the sample matrix with a low background level. To determine absolute MDL, statistically based procedures are available from EPA methods. The MDL studies will be performed annually.

The detection limit is defined as follows for all measurements:

$$MDL = t_{(n-1, 1-\alpha, = 0.99)} \times S$$

where: MDL = method detection limit,  
S = standard deviation of the replicate analyses, and  
 $t_{(n-1, 1-\alpha, = 0.99)}$  = Student's t-value appropriate to a 99-percent confidence level and a standard deviation estimate with n-1 degrees of freedom.

The reporting limits in Section 5.0 are derived from MDLs.

### 11.3.2 PRACTICAL QUANTITATION LIMIT (PQL)

The PQL is defined as 12 times the standard deviation that is derived from the procedures used to determine MDL.

### 11.4 COMPLETENESS

Completeness is not an FDER requirement but is an ESE-required objective.

Completeness is defined by EPA as "a measure of the amount of valid data obtained from a measurement system compared to the amount that was expected to be obtained under correct normal conditions" (EPA, 1980). A completeness of at least 90 percent for each parameter is the objective for this project. Following completion of the analytical testing, percent completeness will be calculated as follows:

$$\text{Completeness (\%)} = \frac{\text{\# of valid y values reported}}{\text{\# of samples collected for analysis of y}} \times 100$$

If completeness is less than 90 percent for any parameter(s), the Project Manager will be notified immediately. The Project Manager is responsible for determining if resampling will be necessary to meet project objectives and will inform the Project QA Officer and Laboratory Coordinator of the decision.

## 12.0 DATA REDUCTION, VALIDATION, AND REPORTING

### 12.1 DATA REDUCTION

Data transfer and reduction are essential functions in summarizing information to support conclusions. It is essential that these processes are performed accurately and, in the case of data reduction, that accepted statistical techniques are used. ESE will use its in-house-developed CLASS™ for data management.

If applicable, example calculations must be included with the analytical method to facilitate review. The entry of input data and calculations should be checked and the signature/initials of the analyst or individual entering the data and reviewer(s) should accompany all data transfers with and without reduction.

For routine analyses performed at the Gainesville Laboratory, sample response data will be entered into CLASS™ by the analyst or other designated individual(s). The computer calculates the following:

1. Linear, quadratic, or logarithmic regression line for standards.
2. Coefficients of variation for replicates.
3. Spiked recoveries.
4. Reference sample concentrations, and
5. Sample concentrations.

Linear or quadratic equations will be used to calculate final data for laboratory analyses requiring a calibration curve:

$$\text{Concentration} = \text{Intercept} + M (\text{Response}) + M2 (\text{Response})^2$$

The equation used to calculate final data is dependent on the linearity of the standard curve and method of analysis.

Purgeable organics by GC/MS are calculated as follows:

$$\text{Concentration } (\mu\text{g/L}) = \frac{(A_{sa})(Q_{is})}{(RF)(A_{is})(PV)}$$

where:  $A_{sa}$  = area from the extracted ion profile of the primary characteristic ion for the target analyte in the sample.

$Q_{is}$  = quantity of the internal standard [nanograms (ng)].

RF = response factor (see Section 8.0).

$A_{is}$  = area from the extracted ion profile of the primary characteristic ion of the internal standard in the sample, and

PV = purge volume (mL).

Semivolatile organics by GC/MS are calculated as follows:

$$\text{Concentration } (\mu\text{g/L}) = \frac{(A_{sa})(Q_{is})}{(A_{is})(RF)} \times \frac{1}{FE} \times \frac{1}{\text{volume}} \times DF$$

where:  $A_{sa}$  = area from the extracted ion profile of the primary characteristic ion for the target analyte in the sample;

$A_{is}$  = area from the extracted ion profile of the primary characteristic ion of the internal standard in the sample;

$Q_{is}$  = quantity of the internal standard (ng);

RF = response factor (see Section 8.0);

FE = fraction extract analyzed =  $\frac{\text{Volume injected } (\mu\text{L})}{\text{extract volume } (\mu\text{L})}$ ;

volume = volume of extracted sample (mL); and

DF = dilution factor =  $\frac{\text{final extract volume for injection (mL)}}{\text{extract volume prior to dilution (mL)}}$ .