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Laboratory Comprehensive Quality Assurance Plan
ENVIRONMENTAL SCIENCE & ENGINEERING, INC.
Peoria Laboratory

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Prepared by:

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Comprehensive Quality Assurance Plan

for
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LIST OF ACRONYMS AND ABBREVIATIC

AAS	atomic absorption spectrophotometry
AIHA	American Industrial Hygiene Association
B	Cyanide, Total and Amenable to Chlorination (Fre
BFB	bromofluorobenzene
BNAs	base/neutrals and acids
BOD	biochemical oxygen demand
BTEX	benzene, toluene, ethylbenzene, xylene
°C	degrees Celsius
CQAP	Comprehensive Quality Assurance Project Plan
CCC	calibration check compounds
CCS	continuing calibration standard
CCV	continuing calibration verification
CFR	Code of Federal Regulations
CLASS™	Chemical Laboratory Analysis and Scheduling Sys
CLP	Contract Laboratory Program
$Cl-CH_2COOH$	chloroacetic acid
COD	chemical oxygen demand
CVAA	*mercury cold vapor atomic absorption*
D	detection limit
DBCP	1,2-Dibromo-3-chloropropane
DFTPP	decafluorotriphenylphosphine
DBASE	data base
DHRS	Department of Health and Rehabilitative Services
DMR-QA	Discharge Monitoring Report - Quality Assurance
DI	deionized

LIST OF ACRONYMS AND ABBREVIAT
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DO	dissolved oxygen
DOT	Department of Transportation
EC	Pesticides/PCBs
ECD	electron capture detector
EDB	1,2-dibromoethane
ELAP	Environmental Laboratory Approval Program
ELPAT	Environmental Lead Proficiency Analytical Testir
EPA	U.S. Environmental Protection Agency
ESE	Environmental Science & Engineering, Inc.
eV	electron volt
FLAA	flame atomic absorption
FID	flame ionization detector
FR	fraction code
FRN	file reference number
ft	foot
g	gram
g/kg	grams per kilogram
GC	gas chromatography
GC/FID	GC employing flame ionization detection
GC/HPLC	gas chromatography/high performance liquid chro
GC/MS	gas chromatograph/mass spectrometer
GC/MS/DS	gas chromatography/mass spectrometry/data syste
GC/NPD	GC employing nitrogen-phosphorus detection
GFAA	graphite furnace atomic absorption
GLP	Good Laboratory Practice

LIST OF ACRONYMS AND ABBREVIATIONS
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GPC	gel permeation chromatography
H	sulfide
HCL	hydrochloric acid
HNO_3	nitric acid
HPLC	high performance liquid chromatography
HWC	hazardous waste coordinator
H_2SO_4	sulfuric acid
IC	ion chromatography
ICAP	inductively coupled argon plasma
ICB	initial calibration blank
ICS	interference check solution
ICV	initial calibration verification
ID	identification
IR	infrared
KCl	potassium chloride
kg	kilogram
KOH	potassium hydroxide
L	liter
LC	Laboratory Coordinator
LIMS	Laboratory Information Management System
MB	method blank
MBAS	methylene blue active substances
MDL	method detection limit
MS	Acid and Base/Neutral Extractables, PNAs, Nitroaromatics
MSDS	Material Safety Data Sheet

LIST OF ACRONYMS AND ABBREVIATIONS
(Continued, Page 4 of 6)

MTBE	methyl-tert-butyl ether
mg/kg	milligrams per kilogram
mg/L	milligrams per liter
mL	milliliter
mm	millimeter
mm^2	square millimeter
NaOH	sodium hydroxide
$Na_2S_2O_3$	sodium thiosulfate
ng	nanogram
N	Metals, Hardness
NIOSH	National Institute of Occupational Safety and Health
NIST	National Institute of Standards and Technology
NPDES	National Pollutant Discharge Elimination System
NTU	nephelometric turbidity unit
O	Oil and Grease, TRPH
PAT	Proficiency Analytical Testing Program
PAH	polynuclear aromatic hydrocarbons
PCB	polychlorinated biphenyl
PCP	pentachlorophenol
PQL	practical quantitation limit
% RSD	percent relative standard deviation
PID	photoionization device
PNA	polynuclear aromatic hydrocarbon
ppb	parts per billion
ppt	parts per thousand
psi	pounds per square inch

LIST OF ACRONYMS AND ABBREVIATIONS
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PVC	polyvinyl chloride
QA	quality assurance
QA/QC	quality assurance/quality control
QAPP	Quality Assurance Project Plan
QC	quality control
RF	response factor
RL	reportable detection limit
RP	replicate
RPD	relative percent difference
RSD	relative standard deviation
S	COD, TOC, Kjeldahl Nitrogen, Ammonia, Total Phosphorus
SD	serial dilution
SOP	standard operating procedure
SOW	Statement of Work
SP	standard spike/ laboratory control sample
SPCC	system performance check compound
SPM	sample matrix spike
SPX	analytical spike
SRT	sample receiving technician
SS	all solids (except VOCs)
STORET	storage and retrieval
SV	volatile solids
SUR	surrogate
THMS	trihalomethanes
TIC	tentatively identified compound
TOC	total organic carbon

LIST OF ACRONYMS AND ABBREVIATIONS
(Continued, Page 6 of 6)

TOX	total organic halides
TRPH	total recoverable petroleum hydrocarbons
TSS	total suspended solids
μg/g	micrograms per gram
μg/L	micrograms per liter
μL	microliter
μmho/cm	micromhos per centimeter
UPS	United Parcel Service
USACE	U.S. Army Corps of Engineers
USEPA	United States Environmental Protection Agency
USGS	U.S. Geological Survey
UV	ultraviolet
V	purgeable compounds
VOA	volatile organic aromatic compound
VOC	volatile organic compound
VP	purgeable aromatics (BTEX)
X	TOX
YSI	Yellow Springs Instruments
Z	total phenols

3.0 STATEMENT OF POLICY

3.1 QUALITY ASSURANCE (QA) STATEMENT OF POLICY

It is the policy of Environmental Science & Engineering, Inc. (ESE), Peoria Laboratory, to maintain an active quality assurance/quality control (QA/QC) program that provides analytical data of known and supportable quality and to ensure a high professional standard in analytical data generated in support of projects undertaken by the staff. An established QA/QC philosophy and program are essential for any organization to consistently produce valid laboratory data. To be valid, data is generated under controlled conditions which do not adversely affect data quality. Data is also interpreted by capable professionals who are trained in appropriate scientific disciplines, maintain a current knowledge of their field, and are experts in the applications for which the data is used. The objectives of the QA/QC program are to estimate the quality of each analytical system including precision, accuracy, and sensitivity sufficient for each project. The QA/QC program also assists in the early recognition of nonconformances which might affect data quality. ESE supports a corporate-wide Quality Education System (QES). All employees are trained in the quality improvement process. The training is supplemented at the department level by instructing employees on the importance of QA/QC and the price of nonconformance.

3.2 SCOPE

This Comprehensive Quality Assurance Plan (CQAP) applies to the analyses of samples received by the Peoria Laboratory. The Peoria Laboratory provides field sample collection when required. In addition, the Peoria Laboratory works with field sampling personnel to ensure that all samples received were collected, preserved, and delivered to the laboratory such that the quality of the analytical results are not adversely affected. All major environmental studies and analyses conducted by ESE Peoria Laboratory for projects under the guidance of client or state/federal government agencies are performed in accordance with this CQAP.

When appropriate, this CQAP is filed with a client and/or regulatory agency, and once approved, is referenced in lieu of the repetitive submission of plans for which only a portion of the information is changed.

3.3 DOCUMENT CONTROL

This CQAP is revised periodically as procedural changes become necessary. Changes are documented by the date of each section. The Peoria Laboratory QA/QC Department keeps a distribution list and assigns a unique number to each copy of the CQAP. When a section is revised, the revision date replaces the original date in the heading code and the table of contents is updated. Copies of the revised sections are provided to each individual on the distribution list.

These procedures apply once the plan has been finalized and implemented. These procedures do not apply to draft documents.

4.0 ORGANIZATION AND RESPONSIBILITIES

4.1 LABORATORY OPERATIONS CAPABILITIES

ESE laboratory operations include the following capabilities:

1. Groundwater and surface water analysis,
2. Soil and sediment analysis,
3. Wastewater analysis,
4. Drum analysis,
5. Tissue analysis, and
6. Underground storage tank analysis.

4.2 KEY PERSONNEL

This section includes ESE's Peoria Laboratory key personnel identified by title, with a brief summary of each individuals responsibilities. An organization chart of the Peoria Laboratory is presented in Figure 4-1 and the ESE Corporate organization chart is presented in Figure 4-2.

4.2.1 LABORATORY OPERATIONS PERSONNEL

4.2.1.1 Analytical Services Director

The Analytical Services Director is responsible for the coordination of analytical services between all laboratories under his authority. The Analytical Services Director is responsible for ensuring that similar systems are used at all laboratories so that data delivered by the different laboratories are similar in format, content and quality. The Analytical Services Director provides budgetary oversight of laboratory operations to verify that required financial controls and accounting procedures are in place, and that financial goals are being met. The Analytical Services Director formulates long-term goals in marketing, facilities, staffing, equipment, and analytical capabilities.

4.2.1.2 Laboratory Manager

The Laboratory Manager is responsible for the overall management of the analytical laboratory, including the appointment and supervision of the Laboratory Information Services Manager, Laboratory Operations Managers, Customer Services Manager, and Laboratory QA/QC Coordinator.

4.2.1.3 Laboratory QA/QC Manager

The Laboratory QA/QC Manager is responsible for the overall management of the three laboratory's QA/QC operations, including the appointment and supervision of the Laboratory QA/QC Coordinators.

4.2.1.4 Laboratory QA/QC Coordinator

The Laboratory QA/QC Coordinator is responsible for coordinating the quality and certification programs of the Peoria Laboratory. The Laboratory QA/QC Coordinator also performs laboratory audits, and maintains QC records for inspection by ESE project management. The Laboratory QA/QC Coordinator provides guidance and coordination to resolve any QA/QC deficiencies and reviews precision, accuracy, and blind samples.

4.2.1.5 Laboratory Information Services Manager

The Laboratory Information Services Manager oversees the Peoria Laboratory's computerized data management system and is responsible for the following:

1. Maintaining ESE's Chemical Laboratory Analysis Scheduling System (CLASS™) (refer to Section 10.1);
2. Approval of all changes made to CLASS™.

4.2.1.6 Laboratory Operations Managers

The Analytical Laboratory Operations Managers are responsible for the overall management of their respective analytical inorganic and organic sections, including the appointment and supervision of their Department Managers. The Customer Services

Manager is responsible for the overall management of the project operations within the laboratory including the appointment and supervision of the Laboratory Project Managers, Laboratory Coordinators, and the Sample Custodian(s).

4.2.1.7 Laboratory Project Manager

The Laboratory Project Manager is responsible for the overall management of the project operations within the Peoria Laboratory. The Laboratory Project Manager acts as a liaison between clients and laboratory operations and is responsible for the following:

1. Coordination of sample analyses to meet project or client objectives;
2. Preparation of analytical reports, including coordination with the Laboratory QA/QC Coordinator and laboratory management to ensure that the data are validated prior to release to the client; and
3. Review of any QA/QC deficiencies reported by the Laboratory Department Manager.

4.2.1.8 Laboratory Coordinator

The Laboratory Coordinator is responsible for the management of individual project tasks within the Peoria Laboratory. The Laboratory Coordinator also acts as a liaison between clients and laboratory operations and has the same responsibilities as the Laboratory Project Manager.

4.2.1.9 Sample Custodian

The Sample Custodian checks in the samples from clients upon receipt by the laboratory. The Sample Custodian compares all samples contained in the shipment to the Chain-of-Custody sheets to ensure that all samples designated on the logsheet have been received. The Sample Custodian will note any special remarks concerning the shipment, log all samples into the Laboratory Information Management System (CLASS™), and deliver the logsheets (Arrival Notices) to the Laboratory Coordinator or Project Manager, and Laboratory Department Managers. The Sample Custodian places samples in appropriate storage areas.

Figure 4-1 ESE PEORIA LABORATORY ORGANIZATION CHART

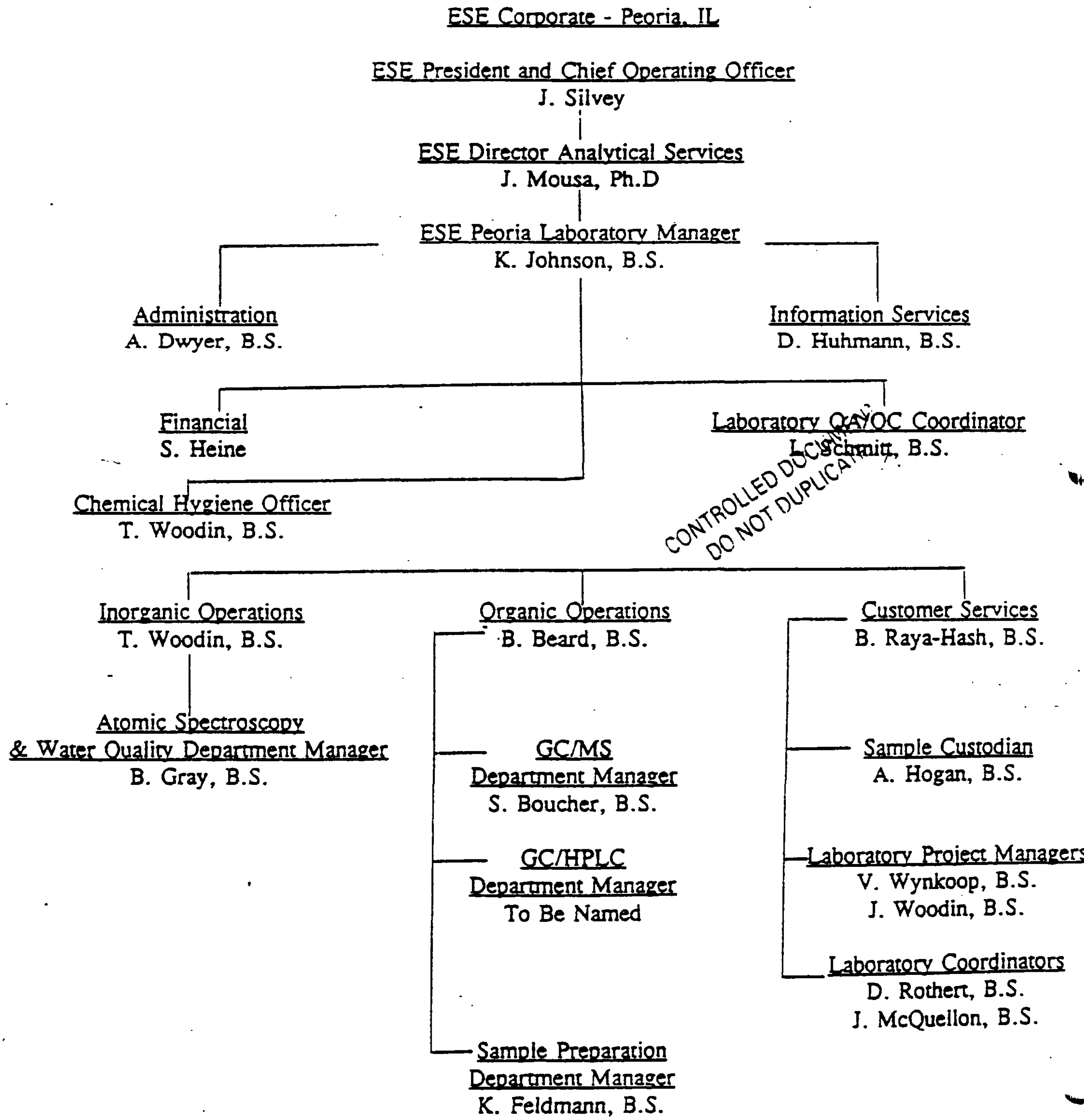

Figure 4.2 ESE CORPORATE ORGANIZATION CHART

Joseph F. Silvey, President and Chief Operating Officer
17390 Brookhurst Street, Suite 110
Fountain Valley, CA 92708
714/964-8722

John J. Mousa, Vice President, Director of Analytical Services, Laboratory Manager
P.O. Box 1703
Gainesville, FL 32602
904/332-3318
800/874-7872

Kim D. Johnson, Peoria Laboratory Manager
8901 North Industrial Road
Peoria, IL 61615
309/692-4422
800/234-1239

4.2.1.10 Laboratory Department Managers

The Laboratory Department Managers are responsible for providing consistent and accurate laboratory data and technical reports produced by their analysts. These individuals are responsible to the Project Manager so to ensure that all personnel under their direction are knowledgeable of the QA/QC requirements of the project and that all QC and technical review procedures are followed and documentation is provided.

4.2.1.11 Laboratory Group Leaders

The Laboratory Group Leaders are responsible for coordinating sample analyses among analysts.

4.2.1.12 Laboratory Analyst

Laboratory Analysts perform laboratory procedures and instrument analyses, including preliminary QC checks to ensure that each batch of data being generated passes all the required QC criteria.

4.2.1.13 Laboratory Chemical Hygiene Officer

The Chemical Hygiene Officer (CHO) will assist laboratory supervisors in implementing the Chemical Hygiene Program. The CHO will provide for Chemical Hygiene Training for analysts, review laboratory safety manual and SOPs, perform safety audits of the laboratory and perform inspections of laboratory safety equipment to determine compliance. Areas of non-compliance will be reported to the appropriate supervisor or manager. The CHO will evaluate worker chemical exposure and will provide a written report of each exposure assessment or determination to the Laboratory Manager for action as necessary.

The Chemical Hygiene Officer monitors all Peoria Laboratory radioactive sources for compliance with ESE and NRC policies on handling radioactive sources. The CHO maintains an inventory of all radioactive sources within the Peoria Laboratory.

5.0 QA OBJECTIVES FOR MEASUREMENT DATA

5.1 LABORATORY ANALYSIS

Analyses are performed according to standard U.S. Environmental Protection Agency (EPA) analytical procedures for analysis of water and soil/sediment unless otherwise specified (Tables 5-1 through 5-55). EPA precision and accuracy data and ESE Laboratory analytical experience were used as the basis for developing criteria to assess laboratory method performance as noted. These limits are subject to change based on actual historic and current performance; updates are provided for insertion into all copies of QAPPs, as appropriate. Limits are updated on a yearly basis unless otherwise specified. Specific compounds are used for controlling purposes in multianalyte methods and are identified in Tables 5-2 through 5-54. Laboratory method performance is evaluated and controlled using calibration checks, blanks, and QC check samples; sample accuracy and precision are evaluated using sample duplicate data, matrix spike, and matrix spike duplicate data. ESE's method control procedures are discussed in Section 11.

The reportable detection limits (RLs) achievable for all parameters are listed in Tables 5-3 through 5-55 (odd numbered tables). The RLs are values, above the method detection limit, which are reported with confidence for typical environmental matrices. The reportable detection limits are not method detection limits (MDLs). Method detection limits are discussed in Section 11. The RLs for waters and those calculated for solids are typically reported as listed, if no matrix and/or other interferences (e.g. salt water) are found to be present (subject to adjustment for dilutions and/or moisture contents).

The following is a brief explanation of the terms and organic method footnotes that appear in Tables 5-1 through 5-57. When recovery criteria was not listed in the method and historical data was not available, the laboratory set achievable QC criteria goals, as noted.

Reference: The reference of the standard analytical methodology used for each procedure.

Precision: Evaluated based on the relative percent difference (RPD) of duplicate spikes (see Section 11 for definition).

Accuracy: Evaluated based on the percent recovery of each spike (see Section 11 for definition).

Units: Volume in liters (L) [e.g., micrograms per liter (μg/L)] indicates a water matrix; control spikes are added to organic-free laboratory water. Mass in grams (g) or kilograms (kg) [e.g., milligrams per kilogram (mg/kg)] indicates a soil/sediment matrix; control spikes are added to blank sample matrices, blank soil, or organic-free laboratory water, depending on the analytical procedure.

Organic Method Footnotes:

a Matrix spike and QC check sample compound.
b Accuracy and precision based on method criteria, unless otherwise noted.
c The QC limits are based on the concentration that can be detected reliably according to ESE Peoria's analytical experience performing the analyses.
d Appendix IX compounds.
e Compound analysis available upon request.
f Compound not listed in method.
g Surrogate compound.
h Estimated detection limits listed in method times a factor of ten.
i Criteria adopted from USEPA Contract Laboratory Program Statement of Work, March 1990.

Table 5-1. Sample Preparation Methods for U.S. EPA SW846 Methods

Sample Preparation Method Number	Description	Matrix	Sample Preparation for Methods
EPA 3005	Acid Digestion	Aqueous	EPA 6010
EPA 3010	Acid Digestion	Aqueous	EPA 6010
EPA 3020	Acid Digestion	Aqueous	EPA 7041, 7060, 7131, 7421, 7740, 7841
EPA 3050	Acid Digestion	Solid	EPA 6010, 7041, 7060, 7131, 7421, 7740, 7841
EPA 3510	Separator Funnel Liquid-Liquid Extraction	Aqueous	EPA 8080, 8141, 8270, 8310
EPA 3520	Continuous Liquid-Liquid Extraction	Aqueous	EPA 8080, 8141, 8270, 8310
EPA 3540	Soxhlet Extraction	Solid	EPA 8080, 8141, 8270, 8310
EPA 3550	Sonication Extraction	Solid	EPA 8080, 8141, 8270, 8310
EPA 5030	Purge-And-Trap	Aqueous, Solid	EPA 8010, 8020, 8240, 8260
EPA 3630	Silica Gel Cleanup	Aqueous, Solid	EPA 8080
EPA 3640	Gel-Permeation Cleanup	Aqueous, Solid	EPA 8080, 8141, 8270
EPA 3660	Sulfur Cleanup	Aqueous, Solid	EPA 8080

Source: ESE.

Table 5-2. Summary of Precision and Accuracy Criteria for Inorganics Analysis, Metals Analysis, Oil and Grease, TRPH, and TOX Analyses

Parameter	Units	Reference	Method Criterion [a] Precision (Max RPD)	Method Criterion [a] Accuracy (Percent Recovery)
Aluminum, Total	µg/L	EPA 200.7, 3005, 3010, 6010	20	80-120
Aluminum, Solid	mg/kg	EPA 3050, 6010	20	80-120
Antimony, Total[b]	µg/L	EPA 204.2, 3020, 7041	20	80-120
Antimony, Total[b]	µg/L	EPA 200.7, 3005, 3010, 6010	20	80-120
Antimony, Solid[b]	mg/kg	EPA 3050, 6010	20	80-120
Antimony, Solid[b]	mg/kg	EPA 3050, 7041	20	80-120
Arsenic, Total[b]	µg/L	EPA 206.2, 200.7, 3005, 3010, 6010, 3020, 7060	20	80-120
Arsenic, Solid[b]	mg/kg	EPA 3050, 7060, 6010	20	80-120
Barium, Total[b]	µg/L	EPA 200.7, 3005, 3010, 6010	20	80-120
Barium, Solid[b]	mg/kg	EPA 3050, 6010	20	80-120
Beryllium, Total[b]	µg/L	EPA 200.7, 3005, 3010, 6010	20	80-120
Beryllium, Solid[b]	mg/kg	EPA 3050, 6010	20	80-120
Cadmium, Total[b]	µg/L	EPA 213.2, 3020, 7131	20	80-120
Cadmium, Total[b]	µg/L	EPA 200.7, 3005, 3010, 6010	20	80-120
Cadmium, Solid[b]	mg/kg	EPA 3050, 6010	20	80-120
Cadmium, Solid[b]	mg/kg	EPA 3050, 7131	20	80-120
Calcium, Total[b]	mg/L	EPA 200.7, 3005, 3010, 6010	20	80-120
Calcium, Solid[b]	mg/kg	EPA 3050, 6010	20	80-120
Chromium, Total[b]	µg/L	EPA 200.7, 3005, 3010, 6010	20	80-120
Chromium, Solid[b]	mg/kg	EPA 3050, 6010	20	80-120
Cobalt, Total[b]	µg/L	EPA 200.7, 3005, 3010, 6010	20	80-120
Cobalt, Solid[b]	mg/kg	EPA 3050, 6010	20	80-120

Table 5-2. *Summary of Precision and Accuracy Criteria for Inorganics Analysis, Metals Analysis, and Oil and Grease, TRPH, and TOX Analyses (Continued, Page 2 of 7)*

Parameter	Units	Reference	Method Criterion [a] Precision (Max RPD)	Accuracy (Percent Recovery)
Copper, Total[b]	µg/L	EPA 200.7, 3005, 3010, 6010	20	80-120
Copper, Solid[b]	mg/kg	EPA 3050, 6010	20	80-120
Iron, Total	µg/L	EPA 200.7, 3005, 3010, 6010	20	80-120
Iron, Solid	mg/kg	EPA 3050, 6010	20	80-120
Lead, Total[b]	µg/L	EPA 239.2, 3020, 7421	20	80-120
Lead, Total[b]	µg/L	EPA 200.7, 3005, 3010, 6010	20	80-120
Lead, Solid[b]	mg/kg	EPA 3050, 6010	20	80-120
Lead, Solid[b]	mg/kg	EPA 3050, 7421	20	80-120
Magnesium, Total	mg/L	EPA 200.7, 3005, 3010, 6010	20	80-120
Magnesium, Solid	mg/kg	EPA 3050, 6010	20	80-120
Manganese, Total	mg/L	EPA 200.7, 3005, 3010, 6010	20	80-120
Manganese, Solid	mg/kg	EPA 3050, 6010	20	80-120
Mercury, Total[b]	µg/L	EPA 245.1, 7470	20	80-120
Mercury, Solid[b]	mg/kg	EPA 7471	20	80-120
Molybdenum, Total	µg/L	EPA 200.7, 3005, 3010, 6010	20	80-120
Molybdenum, Solid	mg/kg	EPA 3050, 6010	20	80-120
Nickel, Total[b]	µg/L	EPA 200.7, 3005, 3010, 6010	20	80-120
Nickel, Solid[b]	mg/kg	EPA 3050, 6010	20	80-120
Potassium, Total	mg/L	EPA 200.7, 3005, 3010, 6010	20	80-120
Potassium, Solid	mg/kg	EPA 3050, 6010	20	80-120

Table 5-2. Summary of Precision and Accuracy Criteria for Inorganics Analysis, Metals Analysis, and Oil and Grease, TRPH, and TOX Analyses (Continued, Page 3 of 7)

			Method Criterion[a]	
Parameter	Units	Reference	Precision (Max RPD)	Accuracy (Percent Recovery)
Selenium, Total[b]	µg/L	EPA 200.7, 3005, 3010, 6010	20	80-120
Selenium, Total[b]	µg/L	EPA 270.2, 3020, 7740	20	80-120
Selenium, Solid[b]	mg/kg	EPA 3050, 7740	20	80-120
Selenium, Solid[b]	mg/kg	EPA 3050, 6010	20	80-120
Silicon, Total	µg/L	EPA 200.7, 3005, 3010, 6010	20	80-120
Silicon, Solid	mg/kg	EPA 3050, 6010	20	80-120
Silver, Total[d]	µg/L	EPA 272.2	20	54-125
Silver, Total[b,d]	µg/L	EPA 200.7, 3005, 3010, 6010	20	54-125
Silver, Solid[b,d]	mg/kg	EPA 3050, 6010	20	54-125
Sodium, Total	mg/L	EPA 200.7, 3005, 3010, 6010	20	80-120
Sodium, Solid	mg/kg	EPA 3050, 6010	20	80-120
Strontium, Total	µg/L	EPA 200.7, 3005, 3010, 6010	20	80-120
Strontium, Solid	mg/kg	EPA 3050, 6010	20	80-120
Thallium, Total[b]	µg/L	EPA 279.2, 3020, 7841	20	80-120
Thallium, Total[b]	µg/L	EPA 200.7, 3005, 3010, 6010	20	80-120
Thallium, Solid[b]	mg/kg	EPA 3050, 6010	20	80-120
Thallium, Solid[b]	mg/kg	EPA 3050, 7841	20	80-120
Titanium, Total	µg/L	EPA 200.7, 3005, 3010, 6010	20	80-120
Titanium, Solid	mg/kg	EPA 3050, 6010	20	80-120
Tin, Total[b]	µg/L	EPA 200.7, 3005, 3010, 6010	20	80-120
Tin, Solid[b]	mg/kg	EPA 3050, 6010	20	80-120
Vanadium, Total[b]	µg/L	EPA 200.7, 3005, 3010, 6010	20	80-120
Vanadium, Solid[b]	mg/kg	EPA 3050, 6010	20	80-120
Zinc, Total[b]	µg/L	EPA 200.7, 3005, 3010, 6010	20	80-120
Zinc, Solid[b]	mg/kg	EPA 3050, 6010	20	80-120

Table 5-2. Summary of Precision and Accuracy Criteria for inorganics Analysis, Metals Analysis, and Oil and Grease, TRPH, and TOX Analyses (Continued, Page 4 of 7)

			Method Criterion [a]	
Parameter	Units	Reference	Precision (Max RPD)	Accuracy (Percent Recovery)
Acidity, Total	mg/L-$CaCO_3$	EPA 305.1	20	80-120
Alkalinity, Total	mg/L-$CaCO_3$	EPA 310.1	20	80-120
BOD, 5-day	mg/L	EPA 405.1	20	80-120
cBOD	mg/L	EPA 405.1	20	80-120
Bromide	mg/L	EPA 320.1, EPA 300, 9056	20	80-120
BTU	Cal/lb	ASTM D-240	N/A	N/A
Carbon, Total	mg/L	EPA 415.2, 9060	20	80-120
Carbon, TOC	mg/L	EPA 415.2, 9060	20	80-120
Carbon, TOC, Solid	mg/kg	EPA 9060 (Mod)	20	80-120
Carbon, Percent Content	% Organic	Walkley Black	N/A	N/A
COD	mg/L	EPA 410.4	20	80-120
Chloride	mg/L	EPA 325.3, 9252, SM 407C	20	80-120
Chloride	mg/L	EPA 300, 9056	20	80-120
Chlorine, Percent	% Chlorine	ASTM D-808	N/A	N/A
Chlorine, Total Residual	mg/L	EPA 330.5	20	80-120
Chromium (+6)	mg/L	EPA 7196	20	80-120
Chromium (+6), Solid	mg/kg	EPA 3060, 7196	20	80-120
Color	Color Units	EPA 110.2	N/A	N/A
Corrosivity	mm/yr	EPA 9040, 1110	N/A	N/A
Cyanide[b]	mg/L	EPA 335.2, 9010	20	80-120
Cyanide, Solid[b]	mg/kg	EPA 9010 (Mod)	20	80-120
Cyanide, Ammenable[b]	mg/L	EPA 335.1	20	80-120
Dissolved Oxygen	mg/L	EPA 360.1	20	N/A
Fluoride	mg/L	EPA 340.2	20	80-120
Fluoride	mg/L	EPA 300, 9056	20	80-120

Table 5-2. Summary of Precision and Accuracy Criteria for Inorganics Analysis, Metals Analysis, and Oil and Grease, TRPH, and TOX Analyses (Continued, Page 5 of 7)

			Method Criterion[a]	
Parameter	Units	Reference	Precision (Max RPD)	Accuracy (Percent Recovery)
Hardness	mg/L-$CaCO_3$	EPA 200.7, 130.2	20	80-120
Ignitability	°C	EPA 1010	N/A	N/A
MBAS (foaming agents)	mg/L	EPA 425.1	20	80-120
Nitrogen, NO_2 + NO_3	mg/L-as N	EPA 353.2	20	80-120
Nitrogen, NO_2 + NO_3	mg/kg-as N	EPA 353.2 (Mod)	20	80-120
Nitrogen, NO_3[e]	mg/L-as N	EPA 300, 9056	20	80-120
Nitrogen, NO_3[e]	mg/L-as N	EPA 353.2	20	80-120
Nitrogen, NO_2	mg/L-as N	EPA 300, 9056	20	80-120
Nitrogen, NO_2	mg/L-as-N	EPA 353.2	20	80-120
Nitrogen, NH_3 + NH_4	mg/L-as N	EPA 350.3	20	80-120
Nitrogen, TKN	mg/L-as N	EPA 351.4	20	80-120
Nitrogen, TKN, Solid	mg/kg-as N	EPA 351.4	20	80-120
Odor, 25°C	Thrsh No	EPA 140.1	N/A	N/A
Oil and Grease, Grav	mg/L	EPA 413.1	20	80-120
Oil and Grease, IR	mg/L	EPA 413.2	20	80-120
Oil and Grease, IR, Solid	mg/kg	EPA 9071	20	80-120
Percent Moisture	% Wet Weight	EPA 160.3	20	N/A
Percent Solids	% Dry Weight	EPA 160.3	20	N/A
pH (including solids)	Std Units	EPA 150.1, 9040	20	N/A
Phenols	mg/L	EPA 420.1	20	80-120
Phenols, Solid	mg/kg	EPA 9065	20	80-120
Phosphorus, Total	mg/L-as P	EPA 365.2	20	80-120
Phosphorus, Ortho	mg/L-as P	EPA 300, 9056	20	80-120
Phosphorus, Ortho	mg/L-as P	EPA 365.2	20	80-120

Table 5-2. Summary of Precision and Accuracy Criteria for Inorganics Analysis, Metals Analysis, and Oil and Grease, TRPH, and TOX Analyses (Continued, Page 6 of 7)

Parameter	Units	Reference	Method Criterion [a] Precision (Max RPD)	Method Criterion [a] Accuracy (Percent Recovery)
Residue, Settleable	mg/L	EPA 160.5	20	N/A
Residue, Susp. (TSS)	mg/L	EPA 160.2	20	N/A
Residue, Diss., Total (TDS) 105°C	mg/L	EPA 160.1	20	N/A
Residue, Total (TS)	mg/L	EPA 160.3	20	N/A
Residue, Volatile	mg/L	EPA 160.4	20	N/A
Petroleum hydrocarbons (TRPH)	mg/L	EPA 418.1	20	80-120
Petroleum hydrocarbons, Solid	mg/kg	EPA 9071/418.1	20	80-120
Silica	mg/L	EPA 200.7	20	80-120
Specific Cond.	µmhos/cm	EPA 120.1	20	N/A
Sulfate	mg/L	EPA 300, 9056	20	80-120
Sulfate	mg/L	EPA 375.4, 9038	20	80-120
Sulfide[b]	mg/L	EPA 376.2, 9030	20	80-120
Sulfide, Solid[b]	mg/kg	EPA 9030	20	80-120
Sulfite	mg/L	EPA 377.1	20	80-120
Sulfur, Percent	% Sulfur	ASTM D-129	N/A	N/A
Temperature	°C	SM 2550 B	N/A	N/A
TOX	µg/L-Cl	EPA 9020A	20	80-120
TOX, Solid	mg/kg	EPA 9020A	20	80-120
Turbidity	NTU	EPA 180.1	20	N/A
TCLP	--	EPA 1311	N/A	N/A

Table 5-2. Summary of Precision and Accuracy Criteria for Inorganics Analysis, Metals Analysis, and Oil and Grease, TRPH, and TOX Analyses (Continued, Page 7 of 7)

Note:

CLP = EPA Contract Laboratory Program.
N/A = not applicable.
SOW = statement of work.
TCLP = toxicity characteristics leaching procedure.
TOX = total organic halides.
TRPH = total recoverable petroleum hydrocarbons.

References:

ASTM D2974—American Society for Testing and Materials Designation: D2974-87, July 1987.
EPA 100-400—Methods for Chemical Analyses of Water and Waste. EPA 600/4-79-020, Revised March 1983.
EPA 1310-9073—Test Methods for Evaluating Solid Waste, SW-846, 3rd Edition, Method 9073, draft 1989: oil and grease methods exclude 7.8 and 7.10).
SM 4500-N—Standard Methods for the Examination of Water and Wastewater, 17th Edition, 1989.

[a] All precision and accuracy criteria is referenced from EPA CLP SOW 3/90.

[b] Appendix IX compounds.

[c] NO_3 (as N) by EPA 353.2 is calculation of (NO_2 + NO_3) - (NO_2); also, method criteria do not apply.

[d] The QC limits are based on the concentration that can be detected reliably according to ESE Peoria's analytical experience performing the analyses.

Source: ESE.

Table 5-3. Reporting Limit Data for Metals, Inorganics, Oil and Grease, TRPH, and TOX Analyses

Parameter	Reference	Reporting Limit: Aqueous[a] (µg/L)	Reporting Limit: Solid[b] (mg/kg)
Aluminum	EPA 200.7, 3005, 3010, 3050, 6010	50	5.0
Antimony	EPA 200.7, 3005, 3010, 3050, 6010	50	5.0
Antimony	EPA 204.2, 3020, 3050, 7041	10	1.0
Arsenic	EPA 200.7, 3005, 3010, 3050, 6010	50	5.0
Arsenic	EPA 206.2, 3020, 3050, 7060	10	1.0
Barium	EPA 200.7, 3005, 3010, 3050, 6010	10	1.0
Beryllium	EPA 200.7, 3005, 3010, 3050, 6010	5.0	0.5
Cadmium	EPA 200.7, 3005, 3010, 3050, 6010	5.0	0.5
Cadmium	EPA 213.2, 3020, 3050, 7131	0.2	0.02
Calcium	EPA 200.7, 3005, 3010, 3050, 6010	500	50
Chromium	EPA 200.7, 3005, 3010, 3050, 6010	10	1.0
Cobalt	EPA 200.7, 3005, 3010, 3050, 6010	10	1.0
Copper	EPA 200.7, 3005, 3010, 3050, 6010	10	1.0
Iron	EPA 200.7, 3005, 3010, 3050, 6010	100	10
Lead	EPA 200.7, 3005, 3010, 3050, 6010	50	5.0
Lead	EPA 239.2, 3020, 3050, 7421	5.0	0.5
Magnesium	EPA 200.7, 3005, 3010, 3050, 6010	500	50
Manganese	EPA 200.7, 3005, 3010, 3050, 6010	10	1.0
Mercury	EPA 245.1, 7470, 7471	0.2	0.02
Molybdenum	EPA 200.7, 3005, 3010, 3050, 6010	50	5.0

Table 5-3. Reporting Limit Data for Metals, Inorganics, Oil and Grease, TRPH, and TOX Analyses (Continued, Page 2 of 5)

Parameter	Reference	Reporting Limit Aqueous[a] (µg/L)	Reporting Limit Solid[b] (mg/kg)
Nickel	EPA 200.7, 3005, 3010, 3050, 6010	20	2.0
Potassium	EPA 200.7, 3005, 3010, 3050, 6010	500	50
Selenium	EPA 200.7, 3005, 3010, 3050, 6010	75	7.5
Selenium	EPA 270.2, 7740, 3020, 3050	5.0	0.5
Silicon	EPA 200.7, 3005, 3010, 3050, 6010	50	5.0
Silver	EPA 200.7, 3005, 3010, 3050, 6010	10	1.0
Silver	EPA 272.2	0.5	0.05
Sodium	EPA 200.7, 3005, 3010, 3050, 6010	500	50
Strontium	EPA 200.7, 6010, 3005, 3010, 3050	10	1.0
Thallium	EPA 200.7, 3005, 3010, 3050, 6010	100	10
Thallium	EPA 279.2, 3020, 3050, 7841	10	1.0
Tin	EPA 200.7, 3005, 3010, 3050, 6010	100	10
Titanium	EPA 200.7, 3005, 3010, 3050, 6010	50	5.0
Vanadium	EPA 200.7, 3005, 3010, 3050, 6010	10	1.0
Zinc	EPA 200.7, 3005, 3010, 3050, 6010	20	2.0

Parameter (Inorganic)	Units	Reference	Reporting Limit[c]
Acidity, Total	mg/L-$CaCO_3$	EPA 305.1	2.0
Alkalinity, Total	mg/L-$CaCO_3$	EPA 310.1	5.0
BOD, 5-day	mg/L	EPA 405.1	1.0
cBOD	mg/L	EPA 405.1	1.0
Bromide	mg/L	EPA 320.1, EPA 300, 9056	0.10
BTU	Cal/lb	ASTM D-240	100
Carbon, Total	mg/L	EPA 415.2, 9060	1.0
Carbon, TOC	mg/L	EPA 415.2, 9060	1.0

Table 5-3. Reporting Limit Data for Metals, Inorganics, Oil and Grease, TRPH, and TOX Analyses (Continued, Page 3 of 5)

Parameter	Units	Reference	Reporting Limit[e]
Carbon, TOC, Solid	mg/kg	EPA 9060 (Mod)	100
Carbon, Percent	% Organic	Walkley Black	0.1
COD	mg/L	EPA 410.4 (Mod)	5.0
Chloride	mg/L	EPA 325.3, 9252, SM 407C	5.0
Chloride	mg/L	EPA 300, 9056	0.5
Chlorine, Percent	% Chlorine	ASTM-D 803	0.1
Chlorine, Total Residual	mg/L	EPA 330.5	0.05
Chromium (+6)	mg/L	EPA 7196	0.05
Chromium (+6) Solid	mg/kg	EPA 3060, 7196	5.0
Color	Color Units	EPA 110.2	N/A
Corrosivity	mm/yr	EPA 9040, 1110	N/A
Cyanide	mg/L	EPA 335.2, 9010	0.005
Cyanide, Solid	mg/kg	EPA 9010 (Mod)	0.50
Cyanide, Ammenable	mg/L	EPA 335.1	0.005
Dissolved Oxygen	mg/L	EPA 360.1	N/A
Fluoride	mg/L	EPA 340.2	0.1
Fluoride	mg/L	EPA 300, 9056	0.5
Hardness	mg/L-$CaCO_3$	EPA 200.7, 130.2	5.0
Ignitability	°C	EPA 1010	N/A
MBAS (foaming agents)	mg/L	EPA 425.1	0.2
Nitrogen, NO_2 + NO_3	mg/L-as N	EPA 353.2	0.10
Nitrogen, NO_2 + NO_3	mg/kg-as N	EPA 353.2 (Mod)	10
Nitrogen, NO_3	mg/L-as N	EPA 300, 9056	0.01
Nitrogen, NO_3	mg/L-as N	EPA 353.2	0.10

Table 5-3. Reporting Limit Data for Metals, Inorganics, Oil and Grease, TRPH, and TOX Analyses (Continued, Page 4 of 5)

Parameter	Units	Reference	Reporting Limit[c]
Nitrogen, NO_2	mg/L-as N	EPA 300, 9056	0.01
Nitrogen, NO_2	mg/L-as N	EPA 353.2	0.05
Nitrogen, NH_3 + NH_4	mg/L-as N	EPA 350.3	0.1
Nitrogen, TKN	mg/L-as N	EPA 351.4	0.1
Nitrogen, TKN, Solid	mg/kg-as N	EPA 351.4	10
Odor, 25°C	Thrsh No	EPA 140.1	N/A
Oil and Grease, Grav	mg/L	EPA 413.1	5.0
Oil and Grease, IR	mg/L	EPA 413.2	1.0
Oil and Grease, IR Solid	mg/kg	EPA 9071	10
Percent Moisture	% Wet Weight	EPA 160.3	N/A
Percent Solids	% Dry Weight	EPA 160.3	N/A
pH (including solids)	Std Units	EPA 150.1, 9040	N/A
Phenols	mg/L	EPA 420.1	0.05
Phenols, Solid	mg/kg	EPA 9065	1.0
Phosphorus, Total	mg/L-as P	EPA 365.2	0.05
Phosphorus, Ortho	mg/L-as P	EPA 365.2	0.05
Phosphorus, Ortho	mg/L-as P	EPA 300, 9056	0.05
Residue, Settleable	mg/L	EPA 160.5	0.1
Residue, Susp. (TSS)	mg/L	EPA 160.2	1.0
Residue, Diss., Total (TDS) 105°C	mg/L	EPA 160.1	1.0
Residue, Total (TS)	mg/L	EPA 160.3	1.0
Residue, Total Volatile	mg/L	EPA 160.4	1.0
Petroleum Hydrocarbons (TRPH)	mg/L	EPA 418.1	1.0

Table 5-3. Reporting Limit Data for Metals, Inorganics, Oil and Grease, TRPH, and TOX Analyses (Continued, Page 5 of 5)

Parameter	Units	Reference	Reporting Limit[c]
Petroleum Hydrocarbons, Solid	mg/kg	EPA 9071/418.1	10
Silica	mg/L	EPA 200.7	2.0
Specific Cond.	μmho/cm	EPA 120.1	10
Sulfate	mg/L	EPA 300, 9056	0.5
Sulfate		EPA 375.4, 9038	5.0
Sulfide	mg/L	EPA 376.2, 9030	0.2
Sulfide, Solid	mg/kg	EPA 9030	5.0
Sulfite	mg/L	EPA 377.1	2.0
Sulfur, Percent	% Sulfur	ASTM D-129	0.1
Temperature	°C	EPA 170.1	N/A
TOX	μg/L-Cl	EPA 9020A	5.0
TOX, Solid	mg/kg	EPA 9020A	10
Turbidity	NTU	EPA 180.1	0.1

Note:
μg/L = micrograms per liter.
mg/kg = milligrams per kilogram.
mg/L = milligrams per liter.
NTU = nephelometric turbidity unit.
μmho/cm = microhms per centimeter.

[a] Based on ESE's instrument detection limit (IDL) studies unless indicated differently. The EPA Contract Laboratory Program (CLP) SOW 3/90 requirements are followed when the IDL studies are conducted.

[b] Based on aqueous IDL studies times a factor of 0.1 to take into account sample weight and final volume of digestate, unless indicated differently.

[c] Based on the lowest standard that ESE routinely uses. For solids, the reporting limits are adjusted for sample weight and final volume.

Source: ESE.

Table 5-4. Analytes, Precision, and Accuracy Data For Volatile Organics, EPA 502.2

Parameter	Aqueous[b] Precision (RPD)	Aqueous[b] Accuracy (% Recovery)
Chloroform[a]	33	77-143
Bromodichloromethane[a]	33	79-137
Dibromochloromethane[a]	33	23-125
Bromoform[a]	33	43-106
THMs, total[a,c]	33	62-128

Reference: EPA Method 502.2 - Volatile Halogenated Compounds in Water by Purge and Trap Gas Chromatography, USEPA, (Revision 2.0) 1989.

[a] Matrix spike and QC check sample compound.
[b] Accuracy and precision based on method criteria, unless otherwise noted.
[c] The QC limits are based on the concentration that can be detected reliably according to ESE Peoria's analytical experience performing the analyses.

Source: ESE.

Table 5-5. Reporting Limit Data For Volatile Organics, EPA 502.2

Parameter	Reporting Limits Aqueous (µg/L)
Chloroform[a]	1.0
Bromodichloromethane[a]	1.0
Dibromochloromethane[a]	1.0
Bromoform[a]	1.0
THMs, total[a]	4.0

[a] Matrix spike and QC check sample compound.

Source: ESE.

Table 5-6. Analytes, Precision, and Accuracy Data For EDB and DBCP, EPA 504

	Aqueous[b]	
	Precision (RPD)	Accuracy (%Recovery)
1,2-Dibromoethane (EDB)[a,d]	20	60-140
DBCP (nemagon)[a,d]	30	60-140

Reference: EPA Method 504- 1,2-Dibromoethane (EDB) and 1,2-Dibromo-3-chloropropane in Water by Microextraction and Gas Chromatography, Methods for the Determination of Organic Compounds in Drinking Water Supplement I, USEPA, July 1990.

[a] Matrix spike and QC check sample compound.
[b] Accuracy and precision criteria based on method criteria, unless otherwise noted.
[d] Appendix IX compounds.

Source: ESE.

Table 5-7. Reporting Limit Data for EDB and DBCP, EPA 504

Parameter	Reporting Limits Aqueous (μg/L)
1,2-Dibromoethane (EDB)[a,d]	0.05
DBCP (nemagon)[a,d]	0.10

[a] Matrix spike and QC check sample compound.
[d] Appendix IX compounds.

Source: ESE.

Table 5-8. Analytes, Precision, and Accuracy Data For Organohalide Pesticides and Aroclors, EPA 505

Parameter	Aqueous [b]	
	Precision (RPD)	Accuracy (%Recovery)
Hexachlorobenzene[a]	19	64-144
Hexachlorocyclopentadiene[a]	22	38-108

Reference: EPA Method 505 - Analysis of Organohalide Pesticides and Commercial Polychlorinated Biphenyl (PCB) Products in Water by Microextraction and Gas Chromatography, <u>Methods for the Determination of Organic Compounds in Drinking Water Supplement I,</u> USEPA, July 1990.

[a] Matrix spike and QC check sample compound.
[b] Accuracy and precision based on method criteria, unless otherwise noted.

Source: ESE.

Table 5-9. Reporting Limits Data for Organohalide Pesticides and Aroclors. EPA 505

Parameter	Reporting Limits Aqueous (µg/L)
Hexachlorobenzene[a]	0.20
Hexachlorocyclopentadiene[a]	0.20

[a] Matrix spike and QC check sample compound.

Source: ESE.

Table 5-10. Analytes, Precision, and Accuracy Data For Phthalate and Adipate Esters, EPA 506

Parameter	Aqueous[b]	
	Precision (RPD)	Accuracy (% Recovery)
Dimethylphthalate	66	25-121
Diethyl phthalate	68	23-119
Di-N-butyl phthalate	66	23-113
Butylbenzyl phthalate	63	26-126
bis (2-Ethylhexyl) adipate[c]	78	15-123
bis (2-Ethylhexyl) phthalate[a,c]	78	15-130
Di-N-octyl phthalate[c]	78	15-131

Reference: EPA Method 506 - Determination of Phthalate and Adipate Esters in Drinking Water by Liquid-Liquid Extraction and Gas Chromatography with Photoionization Detection, Methods for the Determination of Organic Compounds in Drinking Water Supplement I, USEPA, July 1990.

[a] Matrix spike and QC check sample compound.
[b] Accuracy and precision based on method criteria, unless otherwise noted.
[c] The QC limits are based on the concentration that can be detected reliably according to ESE Peoria's analytical experience performing the analyses.

Source: ESE.

Table 5-11. Reporting Limits Data For Phthalate and Adipate Esters, EPA 506

Parameter	Reporting Limits Aqueous (µg/L)
Dimethylphthalate	5.0
Diethyl phthalate	5.0
Di-N-butyl phthalate	5.0
Butylbenzyl phthalate	5.0
bis (2-Ethylhexyl) adipate[a]	5.0
bis (2-Ethylhexyl) phthalate[a]	5.0
Di-N-octyl phthalate	10

[a] Matrix spike and QC check sample compound.

Source: ESE.

Table 5-12. Analytes, Precision, and Accuracy Data For Nitrogen and Phosphorous Containing Pesticides, EPA 507

Parameter	Aqueous[b] Precision (RPD)	Aqueous[b] Accuracy (% Recovery)
Alachlor[a]	34	62-128
Atrazine[a,e]	26	70-130
Bromacil	30	64-118
Butachlor[a]	12	80-112
Butylate	65	34-160
Cyanazine[c,f]	30	70-130
Diazinon	18	92-138
Dyfonate[c,f]	20	80-120
EPTC	32	58-112
Malathion[c,f]	20	80-120
Metolachlor[a,c]	30	70-130
Metribuzin[a,e]	30	70-130
Phorate[c,f]	20	80-120
Propazine	26	68-116
Simazine[a]	21	79-121
Terbufos	12	85-109
Trifluralin[c,f]	30	50-150
2-NMX[c]	N/A	52-115

Table 5-12. Analytes, Precision, and Accuracy Data For Nitrogen and Phosphorous Containing Pesticides, EPA 507 (Continued, Page 2 of 2)

Parameter	Aqueous[b] Precision (RPD)	Accuracy (%Recovery)

Reference: EPA Method 507 - Determination of Nitrogen- and Phosphorous-containing Pesticides in Water by Gas Chromatography with a Nitrogen-Phosphorous Detector, Methods for the Determination of Organic Compounds in Drinking Water Supplement I, USEPA, July 1990.

[a] Matrix spike and QC check sample compound.
[b] Accuracy and precision based on method criteria, unless otherwise noted.
[e] The QC limits are based on the concentration that can be detected reliably according to ESE Peoria's analytical experience performing the analyses.
[f] Compound not listed in method.
[g] Surrogate compound.

Source: ESE.

Table 5-13. Reporting Limit Data for Nitrogen and Phosphorus Containing Pesticides, EPA 507

Parameter	Reporting Limits Aqueous (µg/L)
Alachlor[a]	0.5
Atrazine[a]	0.5
Bromacil[h]	2.5
Butachlor[a,h]	3.8
Butylate	1.5
Cyanazine[f]	1.0
Diazinon	2.5
Dyfonate[f]	0.5
EPTC	2.5
Malathion[f]	0.5
Metolachlor[a,h]	7.5
Metribuzin[a,h]	1.5
Phorate[f]	0.5
Propazine[h]	1.3
Simazine[a,h]	0.75
Terbufos	5.0
Trifluralin[f]	0.5

[a] Matrix spike and QC check sample compound.
[f] Compound not listed in method.
[h] Estimated detection limits listed in EPA Method 507, times ten.

Source: ESE.

Table 5-14. Analytes, Precision, and Accuracy Data For Chlorinated Pesticides, EPA 508

Parameter	Aqueous[b]	
	Precision (RPD)	Accuracy (% Recovery)
BHC,A	33	62-122
BHC,G (Lindane)[a]	33	60-118
Heptachlor[a]	36	63-133
Endosulfan I	30	61-113
Dieldrin[a]	30	61-113
Endrin[a]	30	62-114
DDD	18	88-126
DDT[a]	45	62-162
Methoxychlor[a]	39	64-146
BHC,B	39	64-146
BHC,D	33	58-114
Aldrin[a]	33	58-114
Heptachlor epoxide[a]	30	61-113
Hexachlorobenzene[a]	66	34-164
Chlordane, G	36	63-135
Chlordane, A	36	63-135
DDE	36	63-135
Endosulfan II	33	62-122
Endrin aldehyde	27	64-112
Endosulfan sulfate	45	56-148
Endrin ketone[c,e,f]	30	50-150
Propachlor[a]	27	75-131
Trifluralin[c,e]	30	70-130

Table 5-14. Analytes, Precision, and Accuracy Data For Chlorinated Pesticides, EPA 508 (Continued, Page 2 of 2)

	Aqueous[b]	
	Precision (RPD)	Accuracy (% Recovery)
Toxaphene	20	80-120
Chlordane, Technical	20	80-120
TCX[c,g]	N/A	52-127
DCB[c,g]	N/A	47-148
Aroclor 1016	30	70-130
Aroclor 1254	30	70-130
Aroclor 1260	30	70-130

Reference: EPA Method 508 - Determination of Chlorinated Pesticides in Water by Gas Chromatography with an Electron Capture Detector, Methods for the Determination of Organic Compounds in Drinking Water, USEPA, (Revision 3.0), 1989.

[a] Matrix spike and QC check sample compound.
[b] Accuracy and precision based on method criteria, unless otherwise noted.
[c] The QC limits are based on the concentration that can be detected reliably according to ESE Peoria's analytical experience performing the analyses.
[e] Compound analysis available upon request.
[f] Compound not listed in the method.
[g] Surrogate compound.

Source: ESE.

Table 5-15. Reporting Limit Data For Chlorinated Pesticides, EPA 508

Parameter	Reporting Limits Aqueous (µg/L)
BHC,A	0.05
BHC,G (Lindane)[a]	0.05
Heptachlor[a]	0.05
Endosulfan I	0.05
Dieldrin[a]	0.10
Endrin[a]	0.10
DDD	0.10
DDT[a]	0.10
Methoxychlor[a]	0.50
BHC,B	0.05
BHC,D	0.05
Aldrin[a]	0.05
Heptachlor epoxide[a]	0.05
Hexachlorobenzene[a]	0.05
Chlordane, G	0.50
Chlordane, A	0.50
DDE	0.10
Endosulfan II	0.10
Endrin aldehyde	0.10
Endosulfan sulfate	0.10
Endrin ketone	0.10
Propachlor[a]	0.10
Toxaphene	1.0

Table 5-15. Reporting Limit Data For Chlorinated Pesticides, EPA 508 (Continued, Page 2 of 2)

Parameter	Reporting Limits Aqueous (µg/L)
Chlordane, Technical	1.0
Trifluralin*	0.50
Aroclor 1016	0.50
Aroclor 1254	0.50
Aroclor 1260	0.50

* Matrix spike and QC check sample compound.
* Compound analysis available upon request.

Source: ESE.

Table 5-16. Analytes, Precision, and Accuracy Data For Screening of Polychlorinated Biphenyls. EPA 508A

	Aqueous[b]	
Parameter	Precision (RPD)	Accuracy (% Recovery)
PCBs, as Decachlorobiphenyl	10	80-120

Reference: Screening For Polychlorinated Biphenyls By Perchlorination and Gas Chromatography, Methods for the Determination of Organic Compounds in Drinking Water, USEPA, (Revision 3.0), 1989.

[b] Accuracy and precision based on method criteria, unless otherwise noted.

Source: ESE.

Table 5-17. Reporting Limit Data For Screening of Polychlorinated Biphenyls, EPA 508A

Parameter	Reporting Limits Aqueous (µg/L)
PCBs, as Decachlorobiphenyl	0.1

Source: ESE.

Table 5-18. Analytes, Precision, and Accuracy Data For Chlorinated Herbicides, EPA 515.1

Parameter	Aqueous[b]	
	Precision (RPD)	Accuracy (%Recovery)
2,4-D[a]	63	48-214
2,4,5-TP/Silvex+der.[a]	69	42-226
Dalapon[a,c]	60	30-170
Picloram[a]	52	44-138
Dinoseb[a,c]	72	20-130
Dicamba (banvel)[a]	72	38-232
Pentachlorophenol[a]	72	36-224
2,4,5-T[a]	42	68-166
DCAA[c,g]	N/A	30-115
2,4-DB	30	48-126
Dichlorprop[a]	30	46-168
MCPP[c,e,f]	30	50-150
MCPA[c,e,f]	30	50-150

Reference: EPA Method 515.1 - Determination of Chlorinated Acids in Water by Gas Chromatography with an Electron Capture Detector, Methods for the Determination of Organic Compounds in Drinking Water, USEPA, (Revision 3.0), 1989.

[a] Matrix spike and QC check sample compound.
[b] Accuracy and precision based on method criteria, unless otherwise noted.
[c] The QC limits are based on the concentration that can be detected reliably according the ESE Peoria's analytical experience performing the analyses.
[e] Compound analysis available upon request.
[f] Compound not listed in the method.
[g] Surrogate compound.

Source: ESE.

Table 5-19. Reporting Limit Data For Chlorinated Herbicides, EPA 515.1

Parameter	Reporting Limits Aqueous (µg/L)
2,4-D	2.0
2,4,5-TP/Silvex+der.	1.0
Dalapon	2.0
Picloram	2.0
Dinoseb	2.0
Dicamba (banvel)	1.0
Pentachlorophenol	2.0
2,4-DB	1.0
2,4,5-T	2.0
Dichlorprop	2.0
MCPP[e,f]	200
MCPA[e,f]	200

[a] Matrix spike and QC check sample compound.
[e] Compound analysis available upon request.
[f] Compound not listed in method.

Source: ESE.

Table 5-20. Analytes, Precision, and Accuracy Data for Volatile Organic Compounds, EPA 524.2

Parameter	Aqueous[b] Precision (RPD)	Aqueous[b] Accuracy (% Recovery)
1,1-Dichloroethane	30	80-120
1,2-Dichloroethane[a]	30	80-120
1,1-Dichloroethene[a]	30	80-120
cis-1,2-Dichloroethene[a]	30	80-120
trans-1,2-Dichloroethene[a]	30	80-120
1,2-Dichloropropane[a]	30	80-120
1,3-Dichloropropane	30	80-120
2,2-Dichloropropane	30	80-120
1,1-Dichloropropene	30	80-120
Ethylbenzene[a]	30	80-120
Hexachlorobutadiene	30	80-120
Isopropylbenzene	30	80-120
cis-1,3-Dichloropropene	30	80-120
trans-1,3-Dichloropropene	30	80-120
Naphthalene	30	80-120
Methylene chloride[a]	30	80-120
Styrene[a]	30	80-120
1,1,1,2-Tetrachloroethane	30	80-120
1,1,2,2-Tetrachloroethane	30	80-120
Tetrachloroethene[a]	30	80-120
Toluene[a]	30	80-120
1,2,3-Trichlorobenzene	30	80-120
1,2,4-Trichlorobenzene[a]	30	80-120

Table 5-20. Analytes, Precision, and Accuracy Data for Volatile Organic Compounds, EPA 524.2 (Continued, Page 2 of 3)

Parameter	Aqueous[b] Precision (RPD)	Aqueous[b] Accuracy (% Recovery)
Benzene[a]	30	80-120
Bromobenzene	30	80-120
Bromochloromethane	30	80-120
Bromodichloromethane	30	80-120
Bromoform	30	80-120
Bromomethane	30	80-120
n-Butylbenzene	30	80-120
sec-Butylbenzene	30	80-120
tert-Butylbenzene	30	80-120
Carbon tetrachloride[a]	30	80-120
Chlorobenzene[a]	30	80-120
Chloroform	30	80-120
Chloromethane	30	80-120
Chloroethane	30	80-120
2-Chlorotoluene	30	80-120
4-Chlorotoluene	30	80-120
Dibromochloromethane	30	80-120
4-Isopropyltoluene	30	80-120
n-Propylbenzene	30	80-120
1,2-Dibromo-3-chloropropane	30	80-120
1,2-Dibromoethane	30	80-120
Dibromomethane	30	80-120
1,2-Dichlorobenzene[a]	30	80-120

Table 5-20. Analytes, Precision, and Accuracy Data for Volatile Organic Compounds, EPA 524.2 (Continued, Page 3 of 3)

Parameter	Aqueous[b] Precision (RPD)	Aqueous[b] Accuracy (% Recovery)
1,3-Dichlorobenzene	30	80-120
1,4-Dichlorobenzene[a]	30	80-120
Dichlorodifluoromethane	30	80-120
1,1,1-Trichloroethane[a]	30	80-120
1,1,2-Trichloroethane[a]	30	80-120
Trichloroethene[a]	30	80-120
Trichlorofluoromethane	30	80-120
1,2,3-Trichloropropane	30	80-120
1,2,4-Trimethylbenzene	30	80-120
1,3,5-Trimethylbenzene	30	80-120
Vinyl chloride[a]	30	80-120
Xylenes, total[a]	30	80-120
Dichlorobenzene-D4[c,d]	N/A	50-150
4-Bromofluorobenzene[c,d]	N/A	50-150

Reference: EPA Method 524.2 - Measurement of Purgeable Organic Compounds in Water by Capillary Column Gas Chromatography/Mass Spectrometry, Methods for the Determination of Organic Compounds in Drinking Water, USEPA, (Revision 3.0), 1989.

[a] Matrix spike and QC check sample compound.
[b] Accuracy and precision based on method criteria, unless otherwise noted.
[c] The QC limits are based on the concentration that can be detected reliably according to ESE Peoria's analytical experience performing the analyses.
[d] Surrogate compound.

Source: ESE.

Table 5-21. Reporting Limit Data for Volatile Organic Compounds, EPA 524.2

Parameter	Reporting Limits Aqueous (μg/L)
1,1-Dichloroethane	1.0
1,2-Dichloroethane[a]	0.5
1,1-Dichloroethene[a]	0.5
cis-1,2-Dichloroethene[a]	1.0
trans-1,2-Dichloroethene[a]	1.0
1,2-Dichloropropane[a]	1.0
1,3-Dichloropropane	1.0
2,2-Dichloropropane	1.0
1,1-Dichloropropene	1.0
Ethylbenzene[a]	1.0
Hexachlorobutadiene	1.0
Isopropylbenzene	1.0
cis-1,3-Dichloropropene	1.0
trans-1,3-Dichloropropene	1.0
Naphthalene	1.0
Methylene chloride[a]	1.0
Styrene[a]	1.0
1,1,1,2-Tetrachloroethane	1.0
1,1,2,2-Tetrachloroethane	1.0
Tetrachloroethene[a]	1.0
Toluene[a]	1.0
1,2,3-Trichlorobenzene	1.0
1,2,4-Trichlorobenzene[a]	1.0

Table 5-21. Reporting Limit Data for Volatile Organic Compounds, EPA 524.2 (Continued, Page 2 of 3)

Parameter	Reporting Limits Aqueous (µg/L)
Benzene[a]	0.5
Bromobenzene	1.0
Bromochloromethane	1.0
Bromodichloromethane	1.0
Bromoform	1.0
Bromomethane	2.0
n-Butylbenzene	1.0
sec-Butylbenzene	1.0
tert-Butylbenzene	1.0
Carbon tetrachloride[a]	0.5
Chlorobenzene[a]	1.0
Chloroform	1.0
Chloromethane	2.0
Chloroethane	2.0
2-Chlorotoluene	1.0
4-Chlorotoluene	1.0
Dibromochloromethane	1.0
4-Isopropyltoluene	1.0
n-Propylbenzene	1.0
1,2-Dibromo-3-chloropropane	0.5
1,2-Dibromoethane	0.5
Dibromomethane	1.0
1,2-Dichlorobenzene[a]	1.0

Table 5-21. Reporting Limit Data for Volatile Organic Compounds, EPA 524.2 (Continued, Page 3 of 3)

Parameter	Reporting Limits Aqueous (µg/L)
1,3-Dichlorobenzene	1.0
1,4-Dichlorobenzene[a]	0.5
Dichlorodifluoromethane	2.0
1,1,1-Trichloroethane[a]	0.5
1,1,2-Trichloroethane[a]	1.0
Trichloroethene[a]	1.0
Trichlorofluoromethane	1.0
1,2,3-Trichloropropane	1.0
1,2,4-Trimethylbenzene	1.0
1,3,5-Trimethylbenzene	1.0
Vinyl chloride	0.5
Xylenes, total	1.0

[a] Matrix spike and QC check sample compound.

Source: ESE.

Table 5-22. Analytes, Precision, and Accuracy Data For N-Methylcarbamoxyloximes and N-Methyl Carbamates, EPA 531.1

Parameter	Aqueous[b] Precision (RPD)	Aqueous[b] Accuracy (%Recovery)
Aldicarb[a,e]	9	56-121
Aldicarb sulfone[a,e]	12	68-120
Aldicarb sulfoxide[a,e]	15	59-131
Carbaryl (Sevin)[a]	18	80-114
Carbofuran[a,e]	15	68-119
3-Hydroxycarbofuran[a]	12	90-114
Methomyl[a]	12	92-118
Oxamyl[a]	12	88-112
Methiocarb[a]	30	96-108
Propoxur[a,e,f]	30	70-130

Reference: EPA Method 531.1 - Measurement of n-Methylcarbamoxyloximes and n-Methylcarbamates in Water by Direct Aqueous Injection HPLC with Post Column Derivatization, Methods for the Determination of Organic Compounds in Drinking Water, USEPA, (Revision 3.0), 1989.

[a] Matrix spike and QC check sample compound.
[b] Accuracy and precision criteria based on method, unless otherwise noted.
[e] The QC limits are based on the concentration that can be detected reliably according to ESE Peoria's analytical experience performing the analyses.
[f] Compound not listed in the method.

Source: ESE.

Table 5-23. Reporting Limit Data for N-Methyl Carbamoxyloximes and N-Methyl Carbamates, EPA 531.1

Parameter	Reporting Limits Aqueous (μg/L)
Aldicarb[a]	3.0
Aldicarb sulfone[a]	2.0
Aldicarb sulfoxide[a]	4.0
Carbaryl (Sevin)[a]	10
Carbofuran[a]	40
3-Hydroxycarbofuran[a]	10
Methomyl[a]	10
Oxamyl[a]	10
Methiocarb[e]	10
Propoxur[e,f]	10

[a] Matrix spike and QC check sample compound.
[e] Compound analysis available upon request.
[f] Compound not listed in the method.

Source: ESE.

Table 5-24. Analyte, Precision, and Accuracy Data For Glyphosate, EPA 547

Parameter	Aqueous[b]	
	Precision (RPD)	Accuracy (% Recovery)
Glyphosate	30	70-130

Reference: Determination of Glyphosate in Drinking Water by Direct Aqueous-Injection HPLC, Post Column Derivatization, and Fluorescence Detection, Methods for the Determination of Organic Compounds in Drinking Water-Supplement I, USEPA, July 1990.

[b] Accuracy and precision based on method criteria, unless otherwise noted.

Source: ESE.

Table 5-25. Reporting Limit Data For Glyphosate, EPA 547

Parameter	Reporting Limit Aqueous (µg/L)
Glyphosate	6.0

Source: ESE.

Table 5-26. Analyte, Precision, and Accuracy Data For Diquat, EPA 549[a]

Parameter	Aqueous[b] Precision (RPD)	Aqueous[b] Accuracy (% Recovery)
Diquat	30	70-130

Reference: Determination of Diquat and Paraquat in Drinking Water by Liquid-Solid Extraction and HPLC with Ultraviolet Detection, Methods for the Determination of Organic Compounds in Drinking Water Supplement I, USEPA, July 1990.

[b] Accuracy and precision based on method criteria, unless otherwise noted.

Source: ESE.

Table 5-27. Reporting Limit Data For Diquat, EPA 549

Parameter	Reporting Limit Aqueous (µg/L)
Diquat	0.4

Source: ESE.

Table 5-28. Analytes, Precision, and Accuracy Data For Polycyclic Aromatic Hydrocarbons, EPA 550

Parameter	Aqueous[c] Precision (RPD)	Aqueous[c] Accuracy (% Recovery)
Naphthalene	33	50-110
Acenaphthylene	22	64-110
Acenaphthene	30	60-110
Fluorene	26	62-110
Phenanthrene	43	39-110
Anthracene	13	51-110
Fluoranthene	88	54-126
Pyrene	20	70-110
Benzo(a)anthracene	32	34-118
Chrysene	13	70-118
Benzo(b)flouranthene	32	32-143
Benzo(k)flouranthene	23	66-110
Benzo(a)pyrene[a]	64	46-110
Dibenzo(a,h)anthracene	18	52-110
Benzo(g,h,i)perylene	25	42-120
Indeno(1,2,3-cd)pyrene	12	48-110

Table 5-28. Analytes, Precision, and Accuracy Data For Polycyclic Aromatic Hydrocarbons, EPA 550 (Continued, Page 2 of 2)

Parameter	Aqueous[c] Precision (RPD)	Aqueous[c] Accuracy (% Recovery)
Triphenylene[‡]	N/A	48-140

Reference: EPA Method 550 - Determination of Polycyclic Aromatic Hydrocarbons in Drinking Water by Liquid-Liquid Extraction and HPLC with Coupled Ultraviolet and Fluorescence Detection, Methods for the Determination of Organic Compounds in Drinking Water Supplement I, USEPA, July 1990.

[a] Matrix spike and QC check sample compound.
[c] The QC limits are based on the concentration that can be detected reliably according to ESE Peoria's analytical experience performing the analyses.
[‡] Surrogate compound.

Source: ESE.

Table 5-29. Reporting Limit Data For Polycyclic Aromatic Hydrocarbons, EPA 550

Parameter	Reporting Limits Aqueous (µg/L)
Naphthalene	5.0
Acenaphthylene	5.0
Acenaphthene	5.0
Fluorene	[illegible]
Phenanthrene	0.05
Anthracene	0.05
Fluoranthene	0.05
Pyrene	0.05
Benzo(a)anthracene	0.05
Chrysene	0.05
Benzo(b)flouranthene	0.05
Benzo(k)flouranthene	0.05
Benzo(a)pyrene[a]	0.05
Dibenzo(a,h)anthracene	0.05
Benzo(g,h,i)perylene	0.05
Indeno(1,2,3-cd)pyrene	0.05

[a] Matrix spike and QC check sample compound.

Source: ESE.

Table 5-30. Analytes, Precision, and Accuracy Data For Purgeable Halocarbons, EPA 601 and SW 5030/8010

Parameter	Aqueous[b]		Solid[b]	
	Precision (RPD)	Accuracy (%Recovery)	Precision (RPD)	Accuracy (%Recovery)
Bromodichloromethane[d]	20	42-172	30	42-172
Bromoform[d]	20	13-159	30	13-159
Bromomethane[c,d]	20	15-144	30	15-144
Carbon tetrachloride[d]	20	43-143	30	43-143
Chlorobenzene[a,c,d]	24	71-123	50	38-150
Chloroethane[d]	20	46-137	30	46-137
2-Chloroethylvinyl ether[d]	20	14-186	30	14-186
Chloroform[d]	20	49-133	30	49-133
Chloromethane[c,d]	20	15-190	30	15-190
Dibromochloromethane[c,d]	20	24-190	30	24-190
1,2-Dichlorobenzene[d]	20	37-154	30	37-154
1,3-Dichlorobenzene[d]	20	50-141	30	50-141
1,4-Dichlorobenzene[d]	20	42-143	30	42-143
1,1-Dichloroethane[d]	20	47-132	30	47-132
1,2-Dichloroethane[d]	20	51-147	30	51-147
1,1-Dichloroethene[a,c,d]	38	54-182	30	28-167
trans-1,2-Dichloroethene[d]	20	38-155	30	38-155
1,2-Dichloropropane[d]	20	44-156	30	44-156
cis-1,3-Dichloropropene	20	22-178	30	22-178

Table 5-30. Analytes, Precision, and Accuracy Data for Purgeable Halocarbons, EPA 601 and SW 5030/8010 (Continued, Page 2 of 2)

Parameter	Aqueous[b]		Solid[b]	
	Precision (RPD)	Accuracy (%Recovery)	Precision (RPD)	Accuracy (%Recovery)
trans-1,3-Dichloropropene	20	22-178	30	22-178
Dichlorodifluoromethane[c]	20	70-130	30	70-130
Methylene chloride[d]	20	25-162	30	25-162
1,1,2,2-Tetrachloroethane[d]	20	8-184	30	8-184
Tetrachloroethene[d]	20	26-162	30	26-162
1,1,1-Trichloroethane[d]	20	41-138	30	41-138
1,1,2-Trichloroethane[d]	20	39-136	30	39-136
Trichloroethene[a,c,d]	26	71-123	30	35-146
Trichlorofluoromethane[d]	20	21-156	30	21-156
Vinyl chloride[d]	20	28-163	30	28-163
Bromochloromethane[c,e]	N/A	63-154	N/A	79-115
2-Bromo-1-chloropropane[c,e]	N/A	64-146	N/A	60-114
1,4-Dichlorobutane[c,e]	N/A	68-138	N/A	55-105

Reference: EPA Method SW 8010-- Test Methods for Evaluating Solid Wastes, EPA-SW-846, September 1986.

[a] Matrix spike and QC check sample compound.
[b] Accuracy and precision based on method criteria, unless otherwise noted.
[c] The QC limits are based on the concentration that can be detected reliably according to ESE Peoria's analytical experience performing the analyses.
[d] Appendix IX compounds.
[e] Surrogate compound.

Source: ESE.

Table 5-31. Reporting Limit Data for Purgeable Halocarbons, EPA 601 and SW 5030/8010

Parameter	Reporting Limits Aqueous (μg/L)	Reporting Limits Solid (μg/kg)
Bromodichloromethane[d]	1.0	1.0
Bromoform[d]	5.0	5.0
Bromomethane[d]	5.0	5.0
Carbon tetrachloride[d]	1.0	1.0
Chlorobenzene[a,d]	1.0	1.0
Chloroethane[d]	5.0	5.0
2-Chloroethylvinyl ether[d]	5.0	5.0
Chloroform[d]	1.0	1.0
Chloromethane[d]	5.0	5.0
Dibromochloromethane[d]	1.0	1.0
1,2-Dichlorobenzene[d]	1.0	1.0
1,3-Dichlorobenzene[d]	1.0	1.0
1,4-Dichlorobenzene[d]	1.0	1.0
1,1-Dichloroethane[d]	1.0	1.0
1,2-Dichloroethane[d]	1.0	1.0
1,1-Dichloroethene[a,d]	2.0	2.0
trans-1,2-Dichloroethene[d]	1.0	1.0
1,2-Dichloropropane[d]	5.0	5.0
cis-1,3-Dichloropropene	1.0	1.0
trans-1,3-Dichloropropene	1.0	1.0
Methylene chloride[d]	2.0	2.0
1,1,2,2-Tetrachloroethane[d]	1.0	1.0
Dichlorodifluoromethane	5.0	5.0

Table 5-31. Reporting Limit Data for Purgeable Halocarbons, EPA 601 and SW 5030/8010 (Continued, Page 2 of 2)

	Reporting Limits	
Parameter	Aqueous (µg/L)	Solid (µg/kg)
Tetrachloroethene[d]	1.0	1.0
1,1,1-Trichloroethane[d]	1.0	1.0
1,1,2-Trichloroethane[d]	1.0	1.0
Trichloroethene[a,d]	1.0	1.0
Trichlorofluoromethane[d]	5.0	5.0
Vinyl chloride[d]	5.0	5.0

[a] Matrix spike and QC check sample compound.
[d] Appendix IX compounds.

Source: ESE.

Table 5-32. Analytes, Precision, and Accuracy Data for Purgeable Aromatics, EPA 602 and SW 5030/8020

Parameter	Aqueous[b]		Solid[b]	
	Precision (RPD)	Accuracy (%Recovery)	Precision (RPD)	Accuracy (%Recovery)
Benzene[a,c,d]	20	68-129	30	74-130
Chlorobenzene[d]	20	55-135	30	55-135
1,2-Dichlorobenzene[d]	20	37-154	30	37-154
1,3-Dichlorobenzene[d]	20	50-141	30	50-141
1,4-Dichlorobenzene[d]	20	42-143	30	42-143
Ethylbenzene[d]	20	32-160	30	32-160
Toluene[a,c,d]	20	65-125	30	41-153
Xylenes, total	20	80-126	30	74-128
MTBE[c,f]	20	80-120	30	80-120
Trifluorotoluene[g]	N/A	53-126	N/A	16-130

Reference: EPA Method SW 8020—Test Methods for Evaluating Solid Wastes, EPA-SW-846, September 1986.

MTBE = methyl tert-butyl ether.

[a] Matrix spike and QC check sample compound.
[b] Accuracy and precision based on method criteria, unless otherwise noted.
[c] The QC limits are based on the concentration that can be detected reliably according to ESE Peoria's analytical experience performing the analyses.
[d] Appendix IX compounds.
[f] Compound not listed in the method.
[g] Surrogate compound.

Source: ESE.

Table 5-33. Reporting Limit Data for Purgeable Aromatics, EPA 602 and SW 5030/8020

Parameter	Reporting Limits Aqueous (µg/L)	Solid (µg/kg)
Benzene[a,d]	1.0	1.0
Chlorobenzene[d]	1.0	1.0
1,2-Dichlorobenzene[d]	1.0	1.0
1,3-Dichlorobenzene[d]	1.0	1.0
1,4-Dichlorobenzene[d]	1.0	1.0
Ethylbenzene[d]	1.0	1.0
Toluene[a,d]	1.0	1.0
Xylenes, total	1.0	1.0
MTBE[f]	5.0	5.0

[a] Matrix spike and QC check sample compound.
[d] Appendix IX compounds.
[f] Compound not listed in the method.

Source: ESE.

Table 5-34. Analytes, Precision, and Accuracy Data for Organochlorine Pesticides and PCBs, EPA 608 and SW 3510/3520/3540/3550/8080

Parameter	Aqueous[b] Precision (RPD)	Aqueous[b] Accuracy (%Recovery)	Solid[b] Precision (RPD)	Solid[b] Accuracy (%Recovery)
Aldrin[a,c,d]	30	42-122	50	33-137
BHC,A[d]	30	37-134	50	37-134
BHC,B[d]	30	17-147	50	17-147
BHC,D[d]	30	19-140	50	19-140
BHC, G(lindane)[a,c,d]	30	40-145	50	30-134
Chlordane, A[d]	30	45-119	50	45-119
Chlordane, G[d]	30	45-119	50	45-119
DDD, PP'[d]	30	31-141	50	31-141
DDE, PP'[d]	30	30-145	50	30-145
DDT, PP'[a,c,d]	30	50-140	50	45-145
Dieldrin[a,c,d]	30	53-140	50	44-137
Endosulfan, I[d]	30	45-153	50	45-153
Endosulfan, II[c,d]	30	15-190	50	15-190
Endosulfan sulfate[d]	30	26-144	50	26-144
Endrin[a,c,d]	30	48-143	50	37-153
Endrin aldehyde[c,d]	30	50-160	50	50-160
Endrin ketone[c,e,f]	30	50-160	50	50-160
Heptachlor[a,c,d]	30	44-140	59	30-148
Heptachlor epoxide[d]	30	37-142	50	37-142

Table 5-34. Analytes, Precision, and Accuracy Data for Organochlorine Pesticides and PCBs, EPA 608 and SW 3510/3520/3540/3550/8080 (Continued, Page 2 of 2)

	Aqueous[b]		Solid[b]	
Parameter	Precision (RPD)	Accuracy (%Recovery)	Precision (RPD)	Accuracy (%Recovery)
Methoxychlor[c,d]	30	50-160	50	50-160
Toxaphene[d]	30	41-126	50	41-126
PCB-1016[a,d]	30	50-114	50	50-114
PCB-1221[d]	30	15-178	50	15-178
PCB-1232[c,d]	30	15-190	50	15-190
PCB-1242[d]	30	39-150	50	39-150
PCB 1248[d]	30	38-158	50	38-158
PCB-1254[d]	30	29-131	50	29-131
PCB-1260[a,d]	30	8-127	50	8-127
Mirex[c,e,f]	30	50-160	50	50-160
Trifluralin[c,e,f]	30	50-160	50	50-160
Chlorpyrifos[c,e,f]	30	50-160	50	50-160
Pendimethalin[c,e,f]	30	50-160	50	50-160
Tetrachloro-m-xylene[c,g]	N/A	52-127	N/A	39-119
Decachlorobiphenyl[c,g]	N/A	47-148	N/A	45-127

Reference: EPA Method SW 8080--Test Methods for Evaluating Solid Wastes, EPA-SW-846, September 1986.

[a] Matrix spike and QC check sample compound.
[b] Accuracy and precision based on method criteria, unless otherwise noted.
[c] The QC limits are based on the concentration that can be detected reliably according to ESE Peoria's analytical experience performing the analyses.
[d] Appendix IX compounds.
[e] Compound analysis available upon request.
[f] Compound not listed in the method.
[g] Surrogate compound.

Source: ESE.

Table 5-35. Reporting Limit Data for Organochlorine Pesticides and PCBs, EPA 608 and SW 3510/3520/3540/3550/8080

Parameter	Reporting Limits Aqueous (µg/L)	Reporting Limits Solid (µg/kg)
Aldrin[a,d]	0.05	8.0
BHC,A[d]	0.05	8.0
BHC,B[d]	0.05	8.0
BHC,D[d]	0.05	8.0
BHC, G(lindane)[a,d]	0.05	8.0
Chlordane, A[d]	0.50	80
Chlordane, G[d]	0.50	80
DDD, PP'[d]	0.10	80
DDE, PP'[d]	0.10	16
DDT, PP'[a,d]	0.10	16
Dieldrin[a,d]	0.10	16
Endosulfan, I[d]	0.05	8.0
Endosulfan, II[d]	0.05	8.0
Endosulfan sulfate[d]	0.10	16
Endrin[a,d]	0.10	16
Endrin aldehyde[d]	0.10	16
Endrin ketone[e,f]	0.10	16
Heptachlor[a,d]	0.05	8.0
Heptachlor epoxide[d]	0.05	8.0
Methoxychlor[d]	0.50	80
Toxaphene[d]	1.0	160
Mirex[e,f]	0.10	16
Trifluralin[e,f]	0.05	8.0
Chlorpyrifos[e,f]	0.05	8.0
Pendimethalin[e,f]	0.10	16

Table 5-35. Reporting Limit Data for Organochlorine Pesticides and PCBs, EPA 608 and SW 3510/3520/3540/3550/8080 (Continued, Page 2 of 2)

Parameter	Reporting Limits Aqueous (μg/L)	Solid (μg/kg)
PCB-1016[a,d]	0.50	80
PCB-1221[d]	0.50	80
PCB-1232[d]	0.50	80
PCB-1242[d]	0.50	80
PCB-1248[d]	0.50	80
PCB-1254[d]	1.0	160
PCB-1260[a,d]	1.0	160

[a] Matrix spike and QC check sample compound.
[d] Appendix IX compounds.
[e] Compound analysis available upon request.
[f] Compound not listed in method.

Source: ESE.

Table 5-36. Analytes, Precision, and Accuracy Data for Polynuclear Aromatic Hydrocarbons, EPA 610 and SW 3510/3520/3540/3550/8310

	Aqueous[c]		Solid[c]	
Parameter	Precision (RPD)	Accuracy (%Recovery)	Precision (RPD)	Accuracy (%Recovery)
Acenaphthene[a,d]	15	31-134	50	30-124
Acenaphthylene[d]	30	30-139	50	30-139
Anthracene[d]	30	30-126	50	30-126
Benzo(a)anthracene[d]	30	30-135	50	30-135
Benzo(a)pyrene[d]	30	30-128	50	30-128
Benzo(b)fluoranthene[a,d]	14	30-150	50	30-150
Benzo(ghi)perylene[d]	30	30-116	50	30-116
Benzo(k)fluoranthene[d]	30	30-154	50	30-154
Chrysene[a,d]	16	30-150	50	30-150
Dibenz(a,h)anthracene[d]	30	30-110	50	30-110
Fluoranthene[d]	30	30-123	50	30-123
Fluorene[d]	30	30-142	50	30-142
Indeno(1,2,3-cd)pyrene[d]	30	30-116	50	30-116
Naphthalene[a,d]	16	30-150	50	30-150
Phenanthrene[a,d]	13	30-150	50	30-150
Pyrene[a,d]	16	30-150	50	30-150
Triphenylene[e]	N/A	48-140	N/A	25-133

Reference: EPA Method SW 8310--Test Methods for Evaluating Solid Wastes, EPA-SW-846, September 1986.

[a] Matrix spike and QC check sample compound.
[b] Accuracy and precision based on method criteria, unless otherwise noted.
[c] The QC limits are based on the concentration that can be detected reliably according to ESE Peoria's analytical experience performing the analyses.
[d] Appendix IX compounds.
[e] Surrogate compound.
Source: ESE.

Table 5-37. Reporting Limit Data for Polynuclear Aromatic Hydrocarbons, EPA 610 and SW 3510/3520/3540/3550/8310

Parameter	Reporting Limits Aqueous (μg/L)	Solid (μg/kg)
Acenaphthene[a,d]	10	330
Acenaphthylene[d]	10	330
Anthracene[d]	0.1	3.3
Benzo(a)anthracene[d]	0.1	3.3
Benzo(a)pyrene[d]	0.1	3.3
Benzo(b)fluoranthene[a,d]	0.1	3.3
Benzo(ghi)perylene[d]	0.1	3.3
Benzo(k)fluoranthene[d]	0.1	3.3
Chrysene[a,d]	0.1	3.3
Dibenzo(a,h)anthracene[d]	0.1	3.3
Fluoranthene[d]	0.1	3.3
Fluorene[d]	2.0	70
Indeno(1,2,3-cd)pyrene[d]	0.1	3.3
Naphthalene[a,d]	10	330
Phenanthrene[a,d]	0.1	3.3
Pyrene[a,d]	0.1	3.3

[a] Matrix spike and QC check sample compound.
[d] Appendix IX compounds.

Source: ESE.

Table 5-38. Analytes, Precision, and Accuracy Data for Chlorinated Herbicides, EPA 615 and SW 3510/3520/3540/3550/8150.

Parameter	Aqueous[b] Precision (RPD)	Aqueous[b] Accuracy (%Recovery)	Solid[b] Precision (RPD)	Solid[b] Accuracy (%Recovery)
2,4-D[a,c,d]	30	11-122	50	20-140
2,4-DB	30	84-102	50	84-102
2,4,5-T[a,c,d]	30	67-130	50	67-130
2,4,5-TP/Silvex der.[a,c,d]	30	35-140	50	30-156
Dicamba (banvel)[a]	30	61-113	50	61-113
Dalapon[a,c]	30	42-130	50	42-130
Dichloroprop	30	91-103	50	91-103
Dinoseb[a,c,d]	30	74-130	50	74-130
MCPA	30	86-110	50	86-110
MCPP	30	82-106	50	82-106
Pentachlorophenol[a,c,f]	30	70-130	50	70-130
Picloram[a,c,f]	30	70-130	50	70-130
DCAA[c,g]	N/A	30-130	N/A	30-130

Reference: EPA Method SW 8150—Test Methods for Evaluating Solid Wastes, EPA-SW-846 3rd Edition, September 1986.

[a] Matrix spike and QC check sample compound.
[b] Accuracy and precision based on method criteria, unless otherwise noted.
[c] The QC limits are based on the concentration that can be detected reliably according to ESE Peoria's analytical experience performing the analyses.
[d] Appendix IX compounds.
[f] Compound not listed in method.
[g] Surrogate compound.

Source: ESE.

Table 5-39. Reporting Limit Data for Chlorinated Herbicides, EPA 615 and SW 3510/3520/3540/3550/8150

Parameter	Reporting Limits Aqueous (µg/L)	Reporting Limits Solid (µg/kg)
2,4-D[a,d]	2.0	100
2,4-DB	2.0	100
2,4,5-T[a,d]	1.0	50
2,4,5-TP/Silvex+der.[a,d]	1.0	50
Dicamba (banvel)[a]	1.0	50
Dalapon[a]	2.0	100
Dichloroprop	2.0	100
Dinoseb[a,d]	2.0	100
MCPA	400	20000
MCPP	400	20000
Pentachlorophenol[a,f]	0.2	10
Picloram[a,f]	2.0	100

[a] Matrix spike and QC check sample compound.
[d] Appendix IX compound.
[f] Compound not listed in method.

Source: ESE.

Table 5-40. Analytes, Precision, and Accuracy Data for Organophosphorus Pesticides, EPA 614/622 and SW 3510/3520/3540/3550/8141

Parameter	Aqueous[b] Precision (RPD)	Aqueous[b] Accuracy (%Recovery)	Solid[b] Precision (RPD)	Solid[b] Accuracy (%Recovery)
Bromacil (Hyvar)[c,e,f]	30	50-150	50	50-150
Butachlor (Butanex)[a,c,e,f]	30	50-150	50	50-150
Cyanazine (Bladex)[a,c,f]	30	25-188	50	46-190
Chlorpyrifos (Lorsban)[c]	30	50-150	50	50-150
Demeton[e]	30	36-99	50	36-99
Diazinon (Basudin)[e]	30	49-85	50	49-85
Dichlorvos[e]	30	49-95	50	49-95
Disulfoton (Mocap)[e]	30	55-109	50	55-109
Fonofos (Dyfonate)[c,e,f]	30	50-150	50	50-150
Fenthion (Baytex)[e]	30	9-128	50	9-128
Azinphos methyl (Guthion)[e]	30	16-129	50	16-129
Malathion (Cythion)[c,e]	30	50-150	50	50-150
Metolachlor (Dual or Bicep)[a,c,f]	13	81-105	42	34-136
Parathion ethyl[c,e]	30	50-150	50	50-150
Parathion methyl[e]	30	80-112	50	80-112
Pendimethalin (Prowl)[c,f]	30	50-150	50	50-150
Carbofuran (Furadan)[c,f]	30	50-150	50	50-150
De-ethyl atrazine (DEA)[c,e,f]	30	50-150	50	50-150
De-isopropyl atrazine (DIA)[c,e,f]	30	50-150	50	50-150
Fenchlorphos[c,e,f]	30	50-150	50	50-150
Phorate (Thimet)[e]	30	36-89	50	36-89

Table 5-40. Analytes, Precision, and Accuracy Data for Organophosphorus Pesticides, EPA 614/622 and SW 3510/3520/3540/3550/8141 (Continued, Page 2 of 2)

Parameter	Aqueous[b] Precision (RPD)	Aqueous[b] Accuracy (%Recovery)	Solid[b] Precision (RPD)	Solid[b] Accuracy (%Recovery)
Prometon (Pramitol)[c,f]	30	50-150	50	50-150
Propachlor (Ramrod)[c,e,f]	30	50-150	50	50-150
Propazine (Primatol P)[c,e,f]	30	50-150	50	50-150
Simazine (Princep)[c,e,f]	30	50-150	50	50-150
Alachlor (Lasso)[a,c,f]	34	62-128	43	43-152
Metribuzin (Sencor)[a,c,f]	30	50-150	50	50-150
EPTC(Eptam)[a,c,f]	32	58-112	73	67-190
Butylate (Sutan)[c,f]	30	50-150	50	50-150
Ethalfluralin (Sonalan)[c,e,f]	30	50-150	50	50-150
Trifluralin (Treflan)[a,c,f]	30	50-150	50	50-150
Atrazine (AAtrex)[a,c,f]	26	50-150	44	46-157
Terbufos (Counter)[a,f]	16	79-111	15	88-118
Ethion[c,e]	30	50-150	50	50-150
2-NMX[c,g]	N/A	52-115	N/A	50-150

Reference: EPA Method SW 8141-Test Methods for Evaluating Solid Wastes, EPA-SW-846 3rd Edition, September 1986.

[a] Matrix spike and QC check sample compound.
[b] Accuracy and precision based on method criteria, unless otherwise noted.
[c] The QC limits are based on the concentration that can be detected reliably according to ESE Peoria's analytical experience performing the analyses.
[e] Compound analysis available upon request.
[f] Compound not listed in the method.
[g] Surrogate compound.

Source: ESE.

Table 5-41. Reporting Limit Data for Organophosphorus Pesticides, EPA 614/622 and SW 3510/3520/3540/3550/8141

Parameter	Reporting Limits Aqueous (µg/L)	Solid (µg/kg)
Bromacil (Hyvar)[a,e,f]	1.0	160
Butachlor (Butanex)[a,e,f]	0.50	80
Cyanazine (Bladex)[a,f]	1.0	60
Chlorpyrifos (Lorsban)	0.50	80
Demeton[a]	0.50	80
Diazinon (Basudin)[a]	0.50	80
Dichlorvos[a]	0.50	80
Disulfoton (Mocap)[a]	1.0	160
Fonofos (Dyfonate)[a,f]	0.50	80
Fenthion (Baytex)[a]	0.50	80
Azinphos methyl (Guthion)[a]	1.0	160
Malathion (Cythion)[a]	0.50	80
Metolachlor (Dual or Bicep)[a,f]	0.50	80
Parathion ethyl[a]	0.50	80
Parathion methyl[a]	0.50	80
Pendimethalin (Prowl)[f]	0.50	80
Carbofuran (Furadan)[f]	1.0	160
De-ethyl atrazine (DEA)[a,f]	1.0	160
De-isopropyl atrazine (DIA)[a,f]	1.0	160
Fenchlorphos[a,f]	1.0	160
Phorate (Thimet)[a]	0.50	80
Prometon (Pramitol)[f]	1.0	160
Propachlor (Ramrod)[a,f]	1.0	160
Propazine (Primatol P)[a,f]	0.50	80
Simazine (Princep)[a,f]	0.50	80
Alachlor (Lasso)[a,f]	0.50	80
Metribuzin (Sencor)[a,f]	0.50	80
EPTC (Eptam)[a,f]	0.50	80
Butylate (Sutan)[f]	1.0	160
Ethalfluralin (Sonalan)[e,f]	0.50	80

Table 5-41. Reporting Limit Data for Organophosphorus Pesticides, EPA 614/622 and SW 3510/3520/3540/3550/8141 (Continued, Page 2 of 2)

Parameter	Reporting Limits Aqueous (µg/L)	Solid (µg/kg)
Trifluralin (Treflan)[a,f]	0.50	80
Atrazine (AAtrex)[a,f]	0.50	80
Terbufos (Counter)[e,f]	0.50	80
Ethion[e]	0.50	80

[a] *Matrix spike and QC check sample compound.*
[e] Compound analysis available upon request.
[f] Compound not listed in method.

Source: ESE.

Table 5-42. Analytes, Precision, and Accuracy Data for Volatile Organic Compounds, EPA 624 and SW 5030/8240/8260

	Aqueous[b]		Solid[b]	
Parameter	Precision (RPD)	Accuracy (%Recovery)	Precision (RPD)	Accuracy (%Recovery)
Acetone[c,d,e]	30	61-128	30	61-128
Benzene[a,d,i]	11	76-127	21	66-142
Bromodichloromethane[d]	20	35-155	30	35-155
Bromoform[d]	20	45-169	30	45-169
Bromomethane[c,d]	20	30-190	30	30-190
Carbon tetrachloride[d]	20	70-140	30	70-140
Chlorobenzene[a,d,i]	13	75-130	21	60-133
Chloroethane[c,d]	20	30-190	20	30-190
2-Chloroethylvinyl ether[c]	20	30-190	30	30-190
Chloroform[d]	20	51-138	30	51-138
Chloromethane[c,d]	20	30-190	30	30-190
Dibromochloromethane[d]	20	53-149	30	53-149
1,2-Dichlorobenzene[d]	20	18-190	30	18-190
1,3-Dichlorobenzene[d]	20	59-156	30	59-156
1,4-Dichlorobenzene[d]	20	18-190	20	18-190
1,1-Dichloroethane[d]	20	59-155	30	59-155
1,2-Dichloroethane[d]	20	49-155	30	49-155
1,1-Dichloroethene[a,d,i]	14	61-145	22	59-172
trans-1,2-Dichloroethene[d]	20	54-156	30	54-156

Table 5-42. Analytes, Precision, and Accuracy Data for Volatile Organic Compounds, EPA 624 and SW 5030/8240/8260 (Continued, Page 2 of 4)

Parameter	Aqueous[b] Precision (RPD)	Aqueous[b] Accuracy (%Recovery)	Solid[b] Precision (RPD)	Solid[b] Accuracy (%Recovery)
1,2-Dichloropropane[c,d]	20	30-190	30	30-190
cis-1,3-Dichloropropene[c,d]	20	30-190	30	30-190
trans-1,3-Dichloropropene[d]	20	17-183	30	17-183
Ethyl benzene[d]	20	37-162	20	37-162
Methylene chloride[c,d]	20	30-190	30	30-190
Methyl ethyl ketone (MEK)[c,d,e]	30	60-108	30	60-108
Methyl isobutyl ketone (MIBK)[c,d,e]	30	62-130	30	62-130
Methyl tert butyl ether (MTBE)[c,e]	30	30-190	30	30-190
Styrene[c,d]	30	74-116	30	74-116
1,1,2,2-Tetrachloroethane[d]	20	46-157	30	46-157
Tetrachloroethene[d]	20	64-148	30	64-148
Toluene[a,d,i]	13	76-125	21	59-139
1,1,1-Trichloroethane[d]	20	52-162	30	52-162
1,1,2-Trichloroethane[d]	20	52-150	30	52-150
Trichloroethene[a,d,i]	14	71-120	24	62-137
Trichlorofluoromethane[d]	20	17-181	30	17-181
Vinyl chloride[c,d]	20	30-190	30	30-190
Xylene, total[c,d]	30	58-136	30	58-136
Toluene-D8[g,i]	N/A	88-110	N/A	81-117

Table 5-42. Analytes, Precision, and Accuracy Data for Volatile Organic Compounds, EPA 624 and SW 5030/8240/8260 (Continued, Page 3 of 4)

Parameter	Aqueous[b] Precision (RPD)	Aqueous[b] Accuracy (%Recovery)	Solid[b] Precision (RPD)	Solid[b] Accuracy (%Recovery)
4-Bromofluorobenzene[g,i]	N/A	86-115	N/A	74-121
1,2-Dichloroethane-D4[g,i]	N/A	76-114	N/A	70-121
Acrolein[c,d,e]	30	52-109	30	52-109
Acrylonitrile[c,d,e]	30	70-115	30	70-115
Carbon disulfide[c,d]	30	30-117	30	30-117
Chloroprene[c,d,e,f]	30	30-190	30	30-190
3-Chloropropene[c,d,e,f]	30	30-190	30	30-190
Dichlorodifluoromethane[c,d,e]	30	30-190	30	30-190
trans- 1,4-Dichloro-2-butene[c,d,e,f]	20	69-109	63	30-121
Ethyl Methacrylate[c,d,e]	30	30-190	30	30-190
2-Hexanone[c,d]	30	30-190	30	30-190
n-Hexane[c,e]	30	30-190	30	30-190
Iodomethane[c,d,e]	30	30-190	30	30-190
Methacrylonitrile[c,d,e,f]	30	30-190	30	30-190
cis-1,2-Dichloroethene[e]	30	30-130	30	30-130
2-Butanone[e]	30	30-130	30	30-130
4-Methyl-2-pentanone[e]	30	30-130	30	30-130
Methyl methacrylate[c,d,e,f]	30	30-190	30	30-190
Propionitrile[c,d,e,f]	30	30-190	30	30-190
1.1.1.2-Tetrachloroethane[c,d,e,f]	30	87-125	30	87-125
1.2.3-Trichloropropane[c,d,e]	30	76-125	30	76-125

Table 5-42. Analytes, Precision, and Accuracy Data for Volatile Organic Compounds, EPA 624 and SW 5030/8240/8260 (Continued, Page 4 of 4)

Parameter	Aqueous[b]		Solid[b]	
	Precision (RPD)	Accuracy (%Recovery)	Precision (RPD)	Accuracy (%Recovery)
Vinyl acetate[c,d,e]	30	68-130	30	68-130

Reference: EPA Method SW 8240/8260--Test Methods for Evaluating Solid Wastes, EPA-SW-846 3rd Edition.

[a] Matrix spike and QC check sample compound.
[b] Accuracy and precision based on method criteria, unless otherwise noted.
[c] The QC limits are based on the concentration that can be detected reliably according to ESE Peoria's analytical experience performing the analyses.
[d] Appendix IX compounds.
[e] Compound analysis available upon request.
[f] Compound not listed in method.
[g] Surrogate compound.
[i] Criteria adopted from USEPA Contract Laboratory Program Statement of Work, March 1990.

Source: ESE.

Table 5-43. Reporting Limit Data for Volatile Organic Compounds, EPA 624 and SW 5030/8240/8260

	Reporting Limits	
Parameter	Aqueous (µg/L)	Solid (µg/kg)
Acetone[d,e]	10	10
Benzene[a,d]	5.0	5.0
Bromodichloromethane[d]	5.0	5.0
Bromoform[d]	5.0	5.0
Bromomethane[d]	10	10
Carbon tetrachloride[d]	5.0	5.0
Chlorobenzene[a,d]	5.0	5.0
Chloroethane[d]	10	10
2-Chloroethylvinyl ether	50	50
Chloroform[d]	5.0	5.0
Chloromethane[d]	10	10
Dibromochloromethane[d]	5.0	5.0
1,2-Dichlorobenzene[d]	5.0	5.0
1,3-Dichlorobenzene[d]	5.0	5.0
1,4-Dichlorobenzene[d]	5.0	5.0
1,1-Dichloroethane[d]	5.0	5.0
1,2-Dichloroethane[d]	5.0	5.0
1,1-Dichloroethene[a,d]	5.0	5.0
trans-1,2-Dichloroethene[d]	5.0	5.0
1,2-Dichloropropane[d]	5.0	5.0
cis-1,3-Dichloropropene[d]	5.0	5.0
trans-1,3-Dichloropropene[d]	5.0	5.0
Ethyl benzene[d]	5.0	5.0
Methylene chloride[d]	5.0	5.0
Methyl ethyl ketone[d,e]	10	10
Methyl isobutyl ketone[d,e]	10	10
Methyl tert butyl ether[e]	10	10
Styrene[d]	5.0	5.0

Table 5-43. Reporting Limit Data for Volatile Organic Compounds, EPA 624 and SW 5030/8240/8260 (Continued, Page 2 of 3)

Parameter	Reporting Limits Aqueous (μg/L)	Solid (μg/kg)
1,1,2,2-Tetrachloroethane[d]	5.0	5.0
Tetrachloroethene[d]	5.0	5.0
Toluene[a,d]	5.0	5.0
1,1,1-Trichloroethane[d]	5.0	5.0
1,1,2-Trichloroethane[d]	5.0	5.0
Trichloroethene[a,d]	5.0	5.0
Trichlorofluoromethane[d]	10	10
Vinyl chloride[d]	10	10
Xylene, total[d]	5.0	5.0
Acrolein[d,e]	50	50
Acrylonitrile[d,e]	50	50
Carbon disulfide[d]	5.0	5.0
Chloroprene[d,e,f]	5.0	5.0
3-Chloropropene[d,e,f]	5.0	5.0
Dichlorodifluoromethane[d,e]	10	10
trans-1,4-Dichloro-2-butene[d,e,f]	5.0	5.0
Ethyl methacrylate[d,e]	5.0	5.0
2-Hexanone[d]	10	10
n-Hexane[e]	10	10
Iodomethane[d,e]	5.0	5.0
Methacrylonitrile[d,e,f]	5.0	5.0
Methyl methacrylate[d,e,f]	5.0	5.0
Propionitrile[d,e,f]	5.0	5.0
1,1,1,2-Tetrachloroethane[d,e,f]	5.0	5.0
1,2,3-Trichloropropane[d,e]	5.0	5.0
Vinyl Acetate[d,e]	10	10

Table 5-43. Reporting Limit Data for Volatile Organic Compounds, EPA 624 and SW 5030/8240/8260 (Continued, Page 3 of 3)

Parameter	Reporting Limits Aqueous (μg/L)	Reporting Limits Solid (μg/kg)
cis-1,2-Dichloroethene	5.0	5.0
2-Butanone	5.0	5.0
4-Methyl-2-pentanone	5.0	5.0

[a] Matrix spike and QC check sample compound.
[d] Appendix IX compounds.
[e] Compound analysis available upon request.
[f] Compound not listed in method.

Source: ESE.

Table 5-44. Analytes, Precision, and Accuracy Data for Semivolatile Organic Compounds, EPA 625 and SW 3510/3520/3540/3550/8270

	Aqueous[b]		Solid[b]	
Parameter	Precision (RPD)	Accuracy (%Recovery)	Precision (RPD)	Accuracy (%Recovery)
Acenaphthene[a,d,i]	31	46-118	19	31-137
Acenaphthylene	30	33-145	50	33-145
Anthracene[d]	30	27-133	50	27-133
1,3-Benzenediol[c,d]	30	30-150	50	30-150
Benzidine	30	42-166	50	42-166
Benzo(a)anthracene[d]	30	33-143	50	33-143
Benzo(b)fluoranthene[d]	30	24-159	50	24-159
Benzo(k)fluoranthene[d]	30	11-162	50	11-162
Benzo(a)pyrene[d]	30	17-163	50	17-163
Benzo(ghi)perylene[c,d]	30	30-150	50	30-150
Benzyl alcohol[c,d]	30	30-150	50	30-150
Butylbenzylphthalate[c,d]	30	30-150	50	30-150
bis(2-Chloroethyl)ether[d]	30	12-158	50	12-158
bis(2-Chloroethoxy)-methane[d]	30	33-184	50	33-184
bis(2-Ethylhexyl)-phthalate[c,d]	30	30-158	50	30-158
bis(2-Chloroisopropyl)-ether[d]	30	36-166	50	36-166
4-Bromophenylphenyl-ether[d]	30	53-127	50	53-127
Carabazole[c,e]	30	30-150	50	30-150
2-Chloronaphthalene[d]	30	60-118	50	60-118
2-Chlorophenol[a,d,i]	40	27-123	50	25-102

Table 5-44. Analytes, Precision, and Accuracy Data for Semivolatile Organic Compounds, EPA 625 and SW 3510/3520/3540/3550/8270 (Continued, Page 2 of 7)

Parameter	Aqueous[b] Precision (RPD)	Aqueous[b] Accuracy (%Recovery)	Solid[b] Precision (RPD)	Solid[b] Accuracy (%Recovery)
4-Chloro-3-methylphenol[a,d,i]	42	23-97	33	26-103
4-Chlorophenylphenyl ether[d]	30	25-158	50	25-158
Chrysene[d]	30	17-168	50	17-168
Dibenzo(a,h)anthracene[c,d]	30	30-150	50	30-150
Di-n-butylphthalate[c,d]	30	30-118	50	30-118
1,3-Dichlorobenzene[c,d]	30	30-150	50	30-150
1,2-Dichlorobenzene[d]	30	32-129	50	32-129
1,4-Dichlorobenzene[a,d,i]	28	36-97	27	28-104
3,3'-Dichlorobenzidine[c,d]	30	30-150	50	30-150
2,4-Dichlorophenol[d]	30	39-135	50	39-135
Diethylphthalate[c,d]	30	30-150	50	30-150
2,4-Dimethylphenol[d]	30	32-119	50	32-119
Dimethylpthalate[c,d]	30	30-150	50	30-150
2,4-Dinitrophenol[c,d]	30	30-150	50	30-150
2,4-Dinitrotoluene[a,d,i]	38	24-96	47	28-89
2,6-Dinitrotoluene[d]	30	50-158	50	50-158
Di-n-octylphthalate[c,d]	30	30-146	50	30-146
Fluoranthene[d]	30	26-137	50	26-137
Fluorene[d]	30	59-121	50	59-121
Hexachlorobenzene[c,d]	30	30-150	50	30-150
Hexachlorobutadiene[d]	30	24-116	50	24-116
Hexachlorocyclopentadiene[c,d]	30	50-130	50	50-130

Table 5-44. Analytes, Precision, and Accuracy Data for Semivolatile Organic Compounds, EPA 625 and SW 3510/3520/3540/3550/8270 (Continued, Page 3 of 7)

	Aqueous[b]		Solid[b]	
Parameter	Precision (RPD)	Accuracy (%Recovery)	Precision (RPD)	Accuracy (%Recovery)
Hexachloroethane[d]	30	40-113	50	40-113
Indeno(1,2,3-cd)pyrene[c,d]	30	30-150	50	30-150
Isophorone[d]	30	21-196	50	21-196
2-Methyl-4,6-dinitrophenol[d]	30	30-150	50	30-150
Naphthalene[d]	30	21-133	50	21-133
Nitrobenzene[d]	30	35-180	50	35-180
2-Nitrophenol[d]	30	29-182	50	29-182
4-Nitrophenol[a,d,i]	50	10-80	50	11-114
n-Nitrosodimethylamine[d]	30	52-191	50	32-191
n-Nitrosodi-n-propylamine[a,d,i]	38	41-116	38	41-126
n-Nitrosodiphenylamine[d]	30	40-112	50	40-112
Pentachlorophenol[a,d,i]	50	9-103	47	17-109
Phenanthrene[d]	30	54-120	50	54-120
Phenol[a,d,i]	42	12-110	35	26-90
Pyrene[a,d,i]	31	26-127	36	35-142
1,2,4-Trichlorobenzene[a,d,i]	28	39-98	23	38-107
2,4,6-Trichlorophenol[d]	30	37-144	50	37-144
Acetophenone[c,d,e]	30	10-150	50	10-150
2-Acetylaminofluorene[c,d,e,f]	30	10-150	50	10-150
4-Aminobiphenyl[c,d,e]	30	10-150	50	10-150
Aniline[c,d,e]	30	10-150	50	10-150

Table 5-44. Analytes, Precision, and Accuracy Data for Semivolatile Organic Compounds, EPA 625 and SW 3510/3520/3540/3550/8270 (Continued, Page 4 of 7)

Parameter	Aqueous[b]		Solid[b]	
	Precision (RPD)	Accuracy (%Recovery)	Precision (RPD)	Accuracy (%Recovery)
Aramite[c,d,e,f]	30	10-150	50	10-150
1,4-Benzenediamine[c,d,e,f]	30	10-150	50	10-150
p-Benzoquinone[c,d,e,f]	30	10-150	50	10-150
4-Chloroaniline[c,d,e]	30	10-150	50	10-150
Chlorobenzilate[c,d,e,f]	30	10-150	50	10-150
1-Chloronaphthalene[c,d,e]	30	10-150	50	10-150
Dibenz(a,j)acridine[c,d,e]	30	10-150	50	10-150
Diallate[c,d,e,f]	30	10-150	50	10-150
Dibenzofuran[c,d,e]	30	10-150	50	10-150
2,6-Dichlorophenol[c,d,e]	30	10-150	50	10-150
Dimethoate[c,d,e,f]	30	10-150	50	10-150
p-(Dimethylamino)azo-benzene[c,d,e]	30	10-150	50	10-150
7,12-Dimethylbenz(a) anthracene[c,d,e]	30	10-150	50	10-150
3,3-Dimethylbenzidine[c,d,e,f]	30	10-150	50	10-150
m-Dinitrobenzene[c,d,e,f]	30	10-150	50	10-150
Diphenylamine[c,d,e]	30	10-150	50	10-150
1,2-Diphenylhydrazine[c,d,e]	30	10-150	50	10-150
Ethylmethanesulfonate[c,d,e]	30	10-150	50	10-150
a,a-Dimethylphenylamine[c,d,e]	30	10-150	50	10-150
Hexachlorophene[c,d,e,f]	30	10-150	50	10-150
Hexachloropropene[c,d,e,f]	30	10-150	50	10-150

Table 5-44. Analytes, Precision, and Accuracy Data for Semivolatile Organic Compounds, EPA 625 and SW 3510/3520/3540/3550/8270 (Continued, Page 5 of 7)

Parameter	Aqueous[b] Precision (RPD)	Aqueous[b] Accuracy (%Recovery)	Solid[b] Precision (RPD)	Solid[b] Accuracy (%Recovery)
Isosafrole[c,d,e,f]	30	10-150	50	10-150
Methapyrilene[c,d,e,f]	30	10-150	50	10-150
3-Methylcholanthrene[c,d,e]	30	10-150	50	10-150
Methylmethanesulfonate[c,d,e,f]	30	10-150	50	10-150
2-Methylnaphthalene[c,d,e]	30	10-150	50	10-150
2-Methylphenol(o-Cresol)[c,d,e]	30	10-150	50	10-150
3-Methylphenol(m-Cresol)[c,d,e,f]	30	10-150	50	10-150
4-Methylphenol(p-Cresol)[c,d,e]	30	10-150	50	10-150
1-Naphthylamine[c,d,e]	30	10-150	50	10-150
2-Naphthylamine[c,d,e]	30	10-150	50	10-150
2-Nitroaniline[c,d,e]	30	10-150	50	10-150
3-Nitroaniline[c,d,e]	30	10-150	50	10-150
4-Nitroaniline[c,d,e]	30	10-150	50	10-150
N-Nitrosodiethylamine[c,d,e,f]	30	10-150	50	10-150
N-Nitroso-di-n-butylamine[c,d,e]	30	10-150	50	10-150
N-Nitrosomethyl-ethylamine[c,d,e,f]	30	10-150	50	10-150
N-Nitrosomorpholine[c,d,e,f]	30	10-150	50	10-150
N-Nitrosopiperidine[c,d,e]	30	10-150	50	10-150
4-Nitroquinoline-1-oxide[c,d,e,f]	30	10-150	50	10-150
N-Nitrosopyrrolidine[c,d,e,f]	30	10-150	50	10-150
1,4-Naphthoquinone[c,d,e,f]	30	10-150	50	10-150

Table 5-44. Analytes, Precision, and Accuracy Data for Semivolatile Organic Compounds, EPA 625 and SW 3510/3520/3540/3550/8270 (Continued, Page 6 of 7)

	Aqueous[b]		Solid[b]	
Parameter	Precision (RPD)	Accuracy (%Recovery)	Precision (RPD)	Accuracy (%Recovery)
5-Nitro-o-toluidine[c,d,e,f]	30	10-150	50	10-150
Pentachlorobenzene[c,d,e]	30	10-150	50	10-150
Pentachloronitrobenzene[c,d,e]	30	10-150	50	10-150
Phenacetin[c,d,e]	30	10-150	50	10-150
Phorate[c,d,e]	30	10-150	50	10-150
Parathion[c,d,e]	30	10-150	50	10-150
2-Picoline[c,d,e]	30	10-150	50	10-150
Pronamide[c,d,e]	30	10-150	50	10-150
Pyridine[c,d,e,f]	30	10-150	50	10-150
1,2,4,5-Tetrachloro-benzene[c,d,e]	30	10-150	50	10-150
2,3,4,6-Tetrachlorophenol[c,d,e]	30	10-150	50	10-150
2,4,5-Trichlorophenol[c,d,e]	30	10-150	50	10-150
1,3,5-Trinitrobenzene[c,d,e,f]	30	10-150	50	10-150
o-Toluidine[c,d,e,f]	30	10-150	50	10-150
Safrole[c,d,e,f]	30	10-150	50	10-150
Nitrobenzene-D5[c,g]	N/A	35-114	N/A	23-120
2-Fluorobiphenyl[c,g]	N/A	43-116	N/A	30-115
p-Terphenyl-D4[c,g]	N/A	33-141	N/A	18-137
Phenol-D6[c,g]	N/A	10-110	N/A	24-113
2-Fluorophenol[c,g]	N/A	21-110	N/A	25-121
2,4,6-Tribromophenol[c,g]	N/A	10-123	N/A	19-122

Table 5-44. Analytes, Precision, and Accuracy Data for Semivolatile Organic Compounds, EPA 625 and SW 3510/3520/3540/3550/8270 (Continued, Page 7 of 7)

Parameter	Aqueous[b] Precision (RPD)	Aqueous[b] Accuracy (%Recovery)	Solid[b] Precision (RPD)	Solid[b] Accuracy (%Recovery)
2-Chlorophenol-D4[c,g]	N/A	33-110	N/A	20-130
1,2-Dichlorobenzene-D4[c,g]	N/A	16-110	N/A	20-130
Benzoic acid[c,e]	30	10-150	50	10-150
Dinoseb[c,d,e]	30	10-150	50	10-150
Disulfoton[c,d,e]	30	10-150	50	10-150
Famphur[c,d,e]	30	10-150	50	10-150
Isodrin[c,d,e]	30	10-150	50	10-150
Kepone[c,d,e]	30	10-150	50	10-150
Methyl parathion[c,d,e]	30	10-150	50	10-150
Tetraethyl dithiopyrophosphate (Sulfotepp)[c,d,e]	30	10-150	50	10-150
Thionazine[c,e]	30	10-150	50	10-150
O,O,O-Triethyl phosphorothioate[c,d,e]	30	10-150	50	10-150

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Reference: EPA Method SW 8270--Test Methods for Evaluating Solid Wastes, EPA-SW-846 3rd Edition, September 1986.

[a] Matrix spike and QC check sample compound.
[b] Accuracy and precision based on method criteria, unless otherwise noted.
[c] The QC limits are based on the concentration that can be detected reliably according to ESE Peoria's analytical experience performing the analyses.
[d] Appendix IX compounds.
[e] Compound analysis available upon request.
[f] Compound not listed in method.
[g] Surrogate compound.
[i] Criteria adopted from USEPA Contract Laboratory Program Statement of Work, March 1990.

Source: ESE.

Table 5-45. Reporting Limit Data for Semivolatile Organic Compounds, EPA 625 and SW 3510/3520/3540/3550/8270

Parameter	Reporting Limits Aqueous (μg/L)	Solid (μg/kg)
Acenaphthene[a,d]	10	330
Acenaphthylene	10	330
Anthracene[d]	10	330
1,3-Benzenediol[e]	10	330
Benzidine	50	1600
Benzo(a)anthracene[d]	10	330
Benzo(b)fluoranthene[d]	10	330
Benzo(k)fluoranthere[d]	10	330
Benzo(a)pyrene[d]	10	330
Benzo(ghi)perylene[d]	10	330
Benzyl alcohol[d]	10	330
Butylbenzylphthalate[d]	10	330
bis(2-Chloroethyl)ether[d]	10	330
bis(2-Chloroethoxy)methane[d]	10	330
bis(2-Ethylhexyl)phthalate[d]	10	330
bis(2-Chloroisopropyl)ether[d]	10	330
4-Bromophenylphenyl-ether[d]	10	330
Carabazole[e]	10	330
2-Chloronaphthalene[d]	10	330
2-Chlorophenol[a,d]	10	330
4-Chloro-3-methylphenol[a,d]	10	330
4-Chlorophenylphenyl ether[d]	10	330
Chrysene[d]	10	330
Dibenzo(a,h)anthracene[d]	10	330
Di-n-butylphthalate[d]	10	330
1,3-Dichlorobenzene[d]	10	330
1,2-Dichlorobenzene[d]	10	330
1,4-Dichlorobenzene[a,d]	10	330
3,3'-Dichlorobenzidine[d]	20	660

Table 5-45. Reporting Limit Data for Semivolatile Organic Compounds, EPA 625 and SW 3510/3520/3540/3550/8270 (Continued, Page 2 of 6)

Parameter	Reporting Limits: Aqueous (μg/L)	Reporting Limits: Solid (μg/kg)
2,4-Dichlorophenol[d]	10	330
Diethylphthalate[d]	10	330
2,4-Dimethylphenol[d]	10	330
Dimethylphthalate[d]	10	330
2,4-Dinitrophenol[d]	50	1600
2,4-Dinitrotoluene[a,d]	10	330
2,6-Dinitrotoluene[d]	10	330
Di-n-octylphthalate[d]	10	330
Fluoranthene[d]	10	330
Fluorene[d]	10	330
Hexachlorobenzene[d]	10	330
Hexachlorobutadiene[d]	10	330
Hexachlorocyclopentadiene[d]	10	330
Hexachloroethane[d]	10	330
Indeno(1,2,3-cd)pyrene[d]	10	330
Isophorone[d]	10	330
2-Methyl-4,6-dinitrophenol[d]	50	1600
Naphthalene[d]	10	330
Nitrobenzene[d]	10	330
2-Nitrophenol[d]	10	330
4-Nitrophenol[a,d]	50	1600
n-Nitrosodimethylamine[d]	10	330
n-Nitrosodi-n-propylamine[a,d]	10	330
n-Nitrosodiphenylamine[d]	10	330
Pentachlorophenol[a,d]	50	1600
Phenanthrene[d]	10	330
Phenol[a,d]	10	330
Pyrene[a,d]	10	330
1,2,4-Trichlorobenzene[a,d]	10	330

Table 5-45. Reporting Limit Data for Semivolatile Organic Compounds, EPA 625 and SW 3510/3520/3540/3550/8270 (Continued, Page 3 of 6)

	Reporting Limits	
Parameter	Aqueous (µg/L)	Solid (µg/kg)
2,4,6-Trichlorophenol[d]	10	330
Acetophenone[d,e]	10	330
2-Acetylaminofluorene[d,e,f]	10	330
4-Aminobiphenyl[d,e]	10	330
Aniline[d,e]	10	330
Aramite[d,e,f]	10	330
1,4-Benzenediamine[d,e,f]	10	330
p-Benzoquinone[d,e,f]	10	330
4-Chloroaniline[d,e]	10	330
Chlorobenzilate[d,e,f]	10	330
1-Chloronaphthalene[d,e]	10	330
Dibenz(a,j)acridine[d,e]	10	330
Diallate[d,e,f]	10	330
Dibenzofuran[d,e]	10	330
2,6-Dichlorophenol[d,e]	10	330
Dimethoate[d,e,f]	10	330
p-(Dimethylamino)azobenzene[d,e]	10	330
7,12-Dimethylbenz(a)anthracene[d,e]	10	330
3,3-Dimethylbenzidine[d,e,f]	10	330
m-Dinitrobenzene[d,e,f]	10	330
Diphenylamine[d,e]	10	330
1,2-Diphenylhydrazine[d,e]	10	330
Ethylmethanesulfonate[d,e]	10	330
a,a-Dimethylphenethylamine[d,e]	10	330
Hexachlorophene[d,e,f]	10	330
Hexachloropropene[d,e,f]	10	330
Isosafrole[d,e,f]	10	330
Methapyrilene[d,e,f]	10	330
3-Methylcholanthrene[d,e]	10	330

Table 5-45. Reporting Limit Data for Semivolatile Organic Compounds, EPA 625 and SW 3510/3520/3540/3550/8270 (Continued, Page 4 of 6)

Parameter	Reporting Limits: Aqueous (µg/L)	Reporting Limits: Solid (µg/kg)
Methylmethanesulfonate[d,e,f]	10	330
2-Methylnaphthalene[d,e]	10	330
2-Methylphenol (o-Cresol)[d,e]	10	330
3-Methylphenol (m-Cresol)[d,e]	10	330
4-Methylphenol (p-Cresol)[d,e]	10	330
1-Naphthylamine[d,e]	10	330
2-Naphthylamine[d,e]	10	330
2-Nitroaniline[d,e]	50	1600
3-Nitroaniline[d,e]	50	1600
4-Nitroaniline[d,e]	50	1600
N-Nitrosodiethylamine[d,e,f]	10	330
N-Nitroso-di-n-butylamine[d,e]	10	330
N-Nitrosomethylethylamine[d,e,f]	10	330
N-Nitrosomorpholine[d,e,f]	10	330
N-Nitrosopiperidine[d,e]	10	330
4-Nitroquinoline-1-oxide[d,e,f]	10	330
N-Nitrosopyrrolidine[d,e,f]	10	330
1,4-Naphthoquinone[d,e,f]	10	330
5-Nitro-o-toluidine[d,e,f]	10	330
Pentachlorobenzene[d,e]	10	330
Pentachloronitrobenzene[d,e]	10	330
Phenacetin[d,e]	10	330

Table 5-45. Reporting Limit Data for Semivolatile Organic Compounds, EPA 625 and SW 3510/3520/3540/3550/8270 (Continued, Page 5 of 6)

Parameter	Reporting Limits Aqueous (µg/L)	Solid (µg/kg)
Phorate[d,e]	10	330
Parathion[d,e]	10	330
2-Picoline[d,e]	10	330
Pronamide[d,e]	10	330
Pyridine[d,e,f]	10	330
1,2,4,5-Tetrachlorobenzene[d,e]	10	330
2,4,5-Trichlorophenol[d,e]	50	1600
2,3,4,6-Tetrachlorophenol[d,e]	10	330
1,3,5-Trinitrobenzene[d,e,f]	10	330
o-Toluidine[d,e,f]	10	330
Safrole[d,e,f]	10	330
Benzoic acid[e]	10	330
Dinoseb[d,e]	10	330
Disulfoton[d,e]	10	330
Famphur[d,e]	10	330
Isodrin[d,e]	10	330
Kepone[d,e]	10	330
Methyl parathion[d,e]	10	330
Tetraethyl dithiopyrophosphate (Sulfotepp)[d,e]	10	330

Table 5-45. Reporting Limit Data for Semivolatile Organic Compounds, EPA 625 and SW 3510/3520/3540/3550/8270 (Continued, Page 6 of 6)

Parameter	Reporting Limits Aqueous (µg/L)	Reporting Limits Solid (µg/kg)
Thionazine[e]	10	330
O,O,O-Triethyl phosphorothioate[d,e]	10	330

[a] Matrix spike and QC check sample compound.
[d] Appendix IX compounds.
[e] Compound analysis available upon request.
[f] Compound not listed in method.

Source: ESE.

Table 5-46. Analytes, Precision, and Accuracy Data for Nitroaromatics and Nitroamines by High Performance Liquid Chromatography (HPLC), SW 8330

Parameter	Aqueous[c]		Solid[c]	
	Precision (RPD)	Accuracy (% Recovery)	Precision (RPD)	Accuracy (% Recovery)
HMX	13	84-111	18	80-116
RDX[a]	30	51-111	18	71-107
1,3,5-Trinitrobenzene[a]	28	46-102	25	65-115
1,3-Dinitrobenzene	37	58-132	30	70-130
Methyl-2,4,6-Trinitrophenylnitramine (Tetryl)	21	67-109	46	65-157
Nitrobenzene[a]	32	44-108	24	72-120
2,4,6-Trinitrotoluene[a]	38	48-124	23	72-118
2,4-Dinitrotoluene[a]	21	60-102	19	68-106
2,6-Dinitrotoluene	26	67-119	44	58-146
o-Nitrotoluene	28	53-109	22	70-114
m-Nitrotoluene	48	40-136	48	40-136
p-Nitrotoluene	26	60-112	26	60-112
4-Amino-2,6-dinitrotoluene	30	70-130	30	70-130
2-Amino-4,6-dinitrotoluene	30	70-130	30	70-130
3,4-Dinitrotoluene[c,d]	N/A	30-150	N/A	30-150

Reference: EPA Method SW 8330—Test Methods for Evaluating Solid Wastes, EPA-SW-846 3rd Edition, September 1986.

[a] Matrix spike and QC check sample compound.
[c] The QC limits are based on the concentration that can be detected reliably according to ESE Peoria's analytical experience performing the analyses.
[d] Surrogate compound.

Source: ESE.

Table 5-47. Reporting Limit Data for Nitroaromatics and Nitroamines by High Performance Liquid Chromatography, SW 8330

Parameter	Reporting Limits Aqueous (μg/L)	Solid (μg/kg)
HMX	0.65	650
RDX[a]	0.30	300
1,3,5-Trinitrobenzene[a]	0.25	250
1,3-Dinitrobenzene	0.15	150
Methyl-2,4,6-trinitro-phenylnitramine	0.20	200
Nitrobenzene[a]	0.40	400
2,4,6-Trinitrotoluene[a]	0.20	200
2,4-Dinitrotoluene[a]	0.15	150
2,6-Dinitrotoluene	0.25	250
o-Nitrotoluene	0.7	700
m-Nitrotoluene	1.0	700
p-Nitrotoluene	0.7	1,000
4-Amino-2,6-dinitrotoluene	0.7	1,000
2-Amino-4,6-dinitrotoluene	0.7	1,000

[a] Matrix spike and QC check sample compound.

Source: ESE.

Table 5-48. Analytes, Precision, and Accuracy Data for Nonhalogenated Volatile Organics by Flame Ionization Detector, California Method Modified

Parameter	Aqueous[c]		Solid[c]	
	Precision (RPD)	Accuracy (% Recovery)	Precision (RPD)	Accuracy (% Recovery)
Diesel[a]	30	50-150	50	50-150
Gasoline	30	50-150	50	50-150
Jet Fuel	30	50-150	50	50-150
Unidentified Compound	30	50-150	50	50-150
Mineral Spirits	30	50-150	50	50-150
Motor Oil	30	50-150	50	50-150

Reference: California Method Revision 2.0, August 1991, Southern California Laboratory Hazardous Materials Unit.

[a] Matrix spike and QC check sample compound.
[c] The QC limits are based on the concentration that can be detected reliably according to ESE Peoria's analytical experience performing the analyses.

Source: ESE.

Table 5-49. Reporting Limit Data for Nonhalogenated Volatile Organics by Flame Ionization Detector, California Method Modified

Parameter	Reporting Limits Aqueous (μg/L)	Reporting Limits Solid (μg/kg)
Diesel*	0.5	10
Gasoline	0.5	10
Jet Fuel	0.5	10
Unidentified Compound	0.5	10
Mineral Spirits	0.5	10
Motor Oil	10	160

* Matrix spike and QC check sample compound.

Source: ESE.

Table 5-50. Analytes, Precision, and Accuracy Data for Nonhalogenated Volatile Organics by Flame Ionization Detector, SW 5030/8015 Modified

Parameter [a*]	Aqueous[c] Precision (RPD)	Aqueous[c] Accuracy (% Recovery)	Solid[c] Precision (RPD)	Solid[c] Accuracy (% Recovery)
Methanol[f]	30	50-150	50	50-150
Ethanol	30	50-150	50	50-150
Isopropanol[f]	30	50-150	50	50-150
N-Propanol[f]	30	50-150	50	50-150
N-Butanol[f]	30	50-150	50	50-150
T-Butanol[f]	30	50-150	50	50-150
Isobutanol[d,f]	30	50-150	50	50-150
Isoamyl alcohol[f]	30	50-150	50	50-150
Acetaldehyde[f]	30	50-150	50	50-150
Ethyl ether	30	50-150	50	50-150
Ethyl acetate[f]	30	50-150	50	50-150
1,2-Epoxybutane[f]	30	50-150	50	50-150
2-Methoxyethanol[f]	30	50-150	50	50-150
2-Ethoxyethanol[f]	30	50-150	50	50-150
2-Butoxyethanol[f]	30	50-150	50	50-150
Methyl ethyl ketone (MEK)	30	50-150	50	50-150
1,4-Dioxane[d,f]	30	50-150	50	50-150
Isopropyl acetate[f]	30	50-150	50	50-150
Cyclohexanone[f]	30	50-150	50	50-150
Ethylene glycol[f]	30	50-150	50	50-150
Diethylene glycol[f]	30	50-150	50	50-150
Pentachlorethane[d,f]	30	50-150	50	50-150
Acetonitrile[d,f]	30	50-150	50	50-150

Reference: EPA Method SW 8015--Test Methods for Evaluating Solid Wastes, EPA-SW-846 3rd Edition, September 1986.

[c] The QC limits are based on the concentration that can be detected reliably according to ESE Peoria's analytical experience performing the analyses.

[d] Appendix IX compounds.

[f] Compound not listed in the method.

[a*] The target requested is spiked.

Source: ESE.

Table 5-51. Reporting Limit Data for Nonhalogenated Volatile Organics by Flame Ionization Detector, SW 5030/8015 Modified

Parameter	Reporting Limits Aqueous (mg/L)	Reporting Limits Solid (mg/kg)
Methanol[f]	5.0	5.0
Ethanol	5.0	5.0
Isopropanol[f]	5.0	5.0
N-Propanol[f]	5.0	5.0
N-Butanol[f]	5.0	5.0
T-Butanol[f]	5.0	5.0
Isobutanol[d,f]	5.0	5.0
Isoamyl alcohol[f]	5.0	5.0
Acetaldehyde[f]	5.0	5.0
Ethyl ether	5.0	5.0
Ethyl acetate[f]	10	10
1,2-Epoxybutane[f]	5.0	5.0
2-Methoxyethanol[f]	5.0	5.0
2-Ethoxyethanol[f]	5.0	5.0
2-Butoxyethanol[f]	5.0	5.0
Methyl ethyl ketone (MEK)	5.0	5.0
1,4-Dioxane[d,f]	5.0	5.0
Isopropyl acetate[f]	10	10
Cyclohexanone[f]	5.0	5.0
Ethylene glycol[f]	100	100
Diethylene glycol[f]	100	100
Pentachlorethane[d,f]	5.0	5.0
Acetonitrile[d,f]	5.0	5.0

[d] Appendix IX compound.
[f] Compound not listed in method.

Source: ESE.

Table 5-52. Analytes, Precision, and Accuracy Data for Polynuclear Aromatic Hydrocarbons by Flame Ionization Detector, SW 3510/3520/3540/3550/8100

Parameter [a]	Aqueous[c]		Solid[c]	
	Precision (RPD)	Accuracy (% Recovery)	Precision (RPD)	Accuracy (% Recovery)
Acenaphthene	30	30-150	50	30-150
Acenaphthylene	30	30-150	50	30-150
Anthracene	30	30-150	50	30-150
Benzo(a)anthracene	30	30-150	50	30-150
Benzo(a)pyrene	30	30-150	50	30-150
Benzo(b)fluoranthene	30	30-150	50	30-150
Benzo(k)fluoranthene	30	30-150	50	30-150
Benzo(ghi)perylene	30	30-150	50	30-150
Chrysene	30	30-150	50	30-150
Dibenzo(a,h)anthracene	30	30-150	50	30-150
Fluoranthene	30	30-150	50	30-150
Fluorene	30	30-150	50	30-150
Indeno(1,2,3-cd)pyrene	30	30-150	50	30-150
Naphthalene	30	30-150	50	30-150
Phenanthrene	30	30-150	50	30-150
Pyrene	30	30-150	50	30-150

Reference: EPA Method SW 8100--Test Methods for Evaluating Solid Wastes, EPA-SW-846 3rd Edition, September 1986.

[a] The target requested is spiked.
[c] The QC limits are based on the concentration that can be detected reliably according to ESE Peoria's analytical experience performing the analyses.

Source: ESE.

Table 5-53. Reporting Limit Data for Polynuclear Aromatic Hydrocarbons by Flame Ionization Detector, SW 3510/3520/3540/3550/8100

	Reporting Limits	
Parameter	Aqueous (μg/L)	Solid (μg/kg)
Acenaphthene	10	330
Acenaphthylene	10	330
Anthracene	10	330
Benzo(a)anthracene	10	330
Benzo(a)pyrene	10	330
Benzo(b)fluoranthene	10	330
Benzo(k)fluoranthene	10	330
Benzo(ghi)perylene	10	330
Chrysene	10	330
Dibenzo(a,h)anthracene	10	330
Fluoranthene	10	330
Fluorene	10	330
Indeno(1,2,3-cd)pyrene	10	330
Naphthalene	10	330
Phenanthrene	10	330
Pyrene	10	330

Source: ESE.

Table 5-54. Analytes, Precision, and Accuracy Data for Phenols, SW 3510/3520/3540/3550/8040

Parameter	Aqueous[c]		Solid[c]	
	Precision (RPD)	Accuracy (% Recovery)	Precision (RPD)	Accuracy (% Recovery)
2-sec-butyl-4,6-Dinitrophenol (DNBP)	30	30-150	50	30-150
4-Chloro-3-methylphenol[a]	30	30-150	50	30-150
2-Chlorophenol[a,b]	30	38-126	50	30-150
Cresols (methyl phenols)	30	30-150	50	30-150
2-Cyclohexyl-4,6-dinitrophenol	30	30-150	50	30-150
2,4-Dichlorophenol[b]	30	44-119	50	30-150
2,6-Dichlorophenol	30	30-150	50	30-150
2,4-Dimethylphenol[b]	30	24-118	50	30-150
2,4-Dinitrophenol[b]	30	12-145	50	30-150
2-Methyl-4,6-dinitrophenol[b]	30	30-136	50	30-150
2-Nitrophenol[b]	30	43-117	50	30-150
4-Nitrophenol[a,b]	30	13-110	50	30-150
Pentachlorophenol[a,b]	30	36-134	50	30-150
Phenol[a,b]	30	23-108	50	30-150
Tetrachlorophenols	30	30-150	50	30-150
Trichlorophenols	30	30-150	50	30-150
2,4,6-Trichlorophenol[a,b]	30	53-119	50	30-150
2-Fluorophenol[g]	N/A	30-150	N/A	30-150
2,4,6-Tribromophenol[g]	N/A	30-150	N/A	30-150

Reference: EPA Method SW 8040--Test Methods for Evaluating Solid Wastes, EPA-SW-846 3rd Edition, September 1986.

[a] Matrix spike and QC check sample compound.
[b] Accuracy and precision data based on method criteria, unless otherwise noted.
[c] The QC limits are based on the concentration that can be detected reliably according to ESE Peoria's analytical experience performing the analyses.
[g] Surrogate compound.
Source: ESE.

Table 5-55. Reporting Limit Data for Phenols, SW 3510/3520/3540/3550/8040

Parameter	Reporting Limits Aqueous (µg/L)	Solid (µg/kg)
2-sec-butyl-4,6-Dinitrophenol (DNBP)	5.0	5.0
4-Chloro-3-methylphenol[a]	10	10
2-Chlorophenol[a]	5.0	5.0
Cresols (methyl phenols)	5.0	5.0
2-Cyclohexyl-4,6-dinitrophenol	5.0	5.0
2,4-Dichlorophenol	5.0	5.0
2,6-Dichlorophenol	5.0	5.0
2,4-Dimethylphenol	5.0	5.0
2,4-Dinitrophenol	10	10
2-Methyl-4,6-dinitrophenol	5.0	5.0
2-Nitrophenol	5.0	5.0
4-Nitrophenol[a]	5.0	5.0
Pentachlorophenol[a]	5.0	5.0
Phenol[a]	5.0	5.0
Tetrachlorophenols	5.0	5.0
Trichlorophenols	5.0	5.0
2,4,6-Trichlorophenol[a]	5.0	5.0

Source: ESE.

[a] Matrix spike and QC check sample compound.

6.0 SAMPLE HANDLING PROCEDURES

6.1 INTRODUCTION

The laboratory is able to provide field teams with sampling kits that contain all the required sampling bottles, documents, labels, and preservative solutions as needed for any field sampling effort. Requirements for any field sampling performed by the ESE Peoria Laboratory will be documented in a site specific QAPP. This section of the CQAP details sample handling requirements in the laboratory.

6.2 SAMPLE CONTAINERS CLEANING PROCEDURES

6.2.1 CLEANING PROCEDURES

ESE Peoria uses commercially cleaned sample containers. At a minimum, only Type 200 precleaned sample containers, cleaned according to EPA protocols, and provided with a certificate of cleanliness are used. The certificates are kept on file in the QA/QC office. Table 6-1 summarizes the application of these cleaning procedures. Clean sample containers are stored in a storage shed and a preparation room, both separate from the laboratory.

All sample containers are prepared for shipment in a separate room from the laboratory. 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 the containers are maintained and filed in the QA/QC office. Containers are individually labeled or barcoded by the manufacturer referencing lot numbers. It is not necessary to maintain records of lot numbers used for a particular project.

6.2.2 TYPES OF WATER

Deionized (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

Table 6-1. Sample Container Cleaning Procedures*

LEVEL ONE
Glassware and plasticware receive full EPA quality assurance treatment. Containers are cleaned according to EPA recommended wash procedures and undergo strict quality control analysis. Additional sampling custody seals for bottle closures are included in each case. Each case of containers is then custody sealed - chain-of-custody is intact right from the start. Each container is lot number labeled for traceability to the enclosed certificate of analysis.
CLEANING PROCEDURE A
1. Bottles, liners, and caps are washed in laboratory-grade, nonphosphate detergent.
2. Rinsed three times with distilled water.
3. Rinsed with 1:1 nitric acid.
4. Rinsed three times with ASTM Type 1 organic-free water.
5. Oven-dried for one hour.
6. Rinsed with hexane.
7. Oven-dried for one hour.

Note: Cleaning protocols are applied by commercial supplier.

* Provided by Eagle-Picher, 1993, p. 3.

interest and is relatively free of organic compounds. The water is acceptable for use in the initial rinsing of laboratory glassware. Ultrapure water, used for instrumentation, is defined as ESE Milli-Q water that has been additionally treated through a Milli-Q® treatment system and contains no organic compounds of analytical interest above ESE's routine detection limits.

Water, distilled or deionized, other than ESE-treated water may be used if it is of documented equivalent quality. Commercially available distilled water is used for volatile organic method blanks and trip blanks. The water contains no detectable volatile organic compounds of analytical interest. Documentation is maintained to demonstrate reliability and purity of analyte free water sources.

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 the EPA (40 CFR Part 136). The maximum holding times in Table 6-2 apply to water and soils as noted. If maximum holding times are exceeded, the Project Manager notifies the client and the conversation is documented in the Project Manager's telephone record. Samples that exceed the regulatory holding times will be flagged by the Project Manager or Laboratory Coordinator in the final deliverable. Sample container sizes for water and soil matrices are one liter and 500 mL, respectively, except for VOAs. Sample container sizes for water and soil matrices for VOAs are 44 mL and 60 mL (wide mouth), respectively. (Water samples for VOAs should be collected in duplicate.)

6.3.2 SAMPLE PRESERVATION

Sample preservation is generally performed in the laboratory by means of adding the preservatives to the containers before shipment to the field, unless preservation in the field is requested. Sample containers for volatile analysis (water only) and carbamates are pre-preserved by the manufacturer and are shipped to the field as received from the manufacturer. All preservatives are prepared from reagent grade acids and chemicals.

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 collected. Whatever the field sample type, the field crew must package each sample container to ensure its integrity inside the shipping container. This packaging may include packing materials such as Bubble Wrap® or styrofoam fillers.

Sample containers are typically shipped by bonded courier to the ESE laboratory. Samples are shipped by overnight delivery or as soon as possible after collection (usually daily), with a receiving signature required. Sample receipt and log-in at the Peoria Laboratory is performed by the Sample Custodian, as described in Section 7.3.

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

The Chain-of-Custody forms for the samples in each shipping container are sealed in a plastic Ziploc® bag and taped to the inside of the container. ESE Peoria's policy requires sealing all sample shipping containers with evidence tape prior to shipping.

Samples received by the laboratory that require pH adjustment for preservation are randomly checked to determine that the pH adjustment was made. Sample custodians check the first shipment received each day using unit resolution pH paper. The results are recorded in a logbook. Any problems encountered are reported to the Project Manager or Laboratory Coordinator. Upon client request, additional shipments can be checked for proper preservation techniques.

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

Measurement	Container[1]	Preservation	Maximum Holding Time[2] (Waters and Soils)
Metals			
Chromium VI	P	Cool, 4°C	24 hours[3]
Mercury	P	HNO_3 to pH<2	28 days
Metals, except chromium VI and mercury (filtered and unfiltered)	P	HNO_3 to pH<2	6 months
Inorganic Tests			
Acidity	P, G	Cool, 4°C	14 days[3]
Alkalinity	P, G	Cool, 4°C	14 days[3]
Ammonia	P, G	Cool, 4°C, H_2SO_4 to pH<2	28 days[3]
BOD	P, G	Cool, 4°C	48 hours[3]
Bromide	P, G	Cool, 4°C	28 days[3]
BOD, carbonaceous	P, G	Cool, 4°C	48 hours[3]
COD	P, G	Cool, 4°C, H_2SO_4 to pH<2	28 days[3]
Chloride	P, G	Cool, 4°C	28 days[3]
Chlorine, total	P, G	Cool, 4°C	Analyze immediately[3,7]
Color	P, G	Cool, 4°C	48 hours[3]
Cyanide, total and amenable to chlorination	P, G	Cool, 4°C, NaOH to pH>12, 0.6 g ascorbic acid[6]	14 days[3,8]
Fluoride	P	Cool, 4°C	28 days[3]
Hardness	P, G	HNO_3 to pH<2	6 months[3]
Hydrogen ion (pH)	P, G	Cool, 4°C	Analyze immediately[3]
Ignitibility	G	Cool, 4°C	28 days
Kjeldahl and organic nitrogen	P,G	Cool, 4°C, H_2SO_4 to pH<2	28 days[3]
Nitrate	P, G	Cool, 4°C	48 hours[3]
Nitrate-nitrite	P, G	Cool, 4°C, H_2SO_4 to pH<2	28 days[3]
Nitrite	P, G	Cool, 4°C	48 hours[3]
Odor	P, G	Cool, 4°C	24 hours[3]
Oil and grease	G	Cool, 4°C, H_2SO_4 to pH<2	28 days[3]
Organic carbon	P, G	Cool, 4°C, H_2SO_4 to pH<2	28 days[3]
Orthophosphate	P, G	Filter immediately, Cool, 4°C	48 hours[3]
Petroleum Hydrocarbons (TRPH)	G	Cool, 4°C, H_2SO_4 to pH<2	28 days[3]
Phenols	G	Cool, 4°C, H_2SO_4 to pH<2	28 days[3]
Phosphorus (elemental)	G	Cool, 4°C	48 hours[3]
Phosphorus, total	P, G	Cool, 4°C, H_2SO_4 to pH<2	28 days[3]
MBAS	P, G	Cool, 4°C	48 hours[3]
Bromates (IC)	P, G	Cool, 4°C	30 days[3]
Corrosivity (calculated)	P, G	Cool, 4°C	7 days[9]
Residue, total	P, G	Cool, 4°C	7 days[3]
Residue, filterable	P, G	Cool, 4°C	7 days[3]
Residue, nonfilterable (TSS)	P, G	Cool, 4°C	7 days[3]
Residue, settleable	P, G	Cool, 4°C	48 hours[3]
Residue, volatile	P, G	Cool, 4°C	7 days[3]
Silica	P	Cool, 4°C	28 days[3]
Specific conductance	P, G	Cool, 4°C	28 days[3]
Sulfate	P, G	Cool, 4°C	28 days[3]
Sulfide	P, G	Cool, 4°C, add 2 mL zinc acetate plus NaOH to pH>9	7 days[3]
Sulfite	P, G	Cool, 4°C	Analyze immediately[3]

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

Measurement	Container[1]	Preservation	Maximum Holding Time[2] (Waters and Soils)
Temperature	P, G	Cool, 4°C	Analyze immediately[3]
Turbidity	P, G	Cool, 4°C	48 hours[3]
<u>Organic Tests</u>			
Carbamates	G, PTFE-faced silicone septum	Cool, 4°C Cl-CH_2COOH to pH <3	28 days
Glyphosate	G, Teflon®-lined cap	Cool, 4°C *0.008% $Na_2S_2O_3$* store in dark	14 days
Purgeable halocarbons	G, Teflon®-lined septum	Cool, 4°C, 0.008% $Na_2S_2O_3$[5,6] store in dark	14 days
Purgeable aromatic hydrocarbons	G, Teflon®-lined septum	Cool, 4°C, 0.008% $Na_2S_2O_3$[5,6] HCl to pH <2	14 days
Phenols	G, Teflon®-lined cap	Cool, 4°C, 0.008% $Na_2S_2O_3$[5] store in dark	7/40 days for waters[4] 14/40 days for soils[4]
Phthalate esters	G, Teflon®-lined cap	Cool, 4°C, store in dark	7/40 days for waters[4] 14/40 days for soils[4]
PCBs, pesticides, herbicides	G, Teflon®-lined cap	Cool, 4°C, 0.008% NA_2S_2O3[5] store in dark	7/40 days for waters[4] 14/40 days for soils[4]
Polynuclear aromatic hydrocarbons	G, Teflon®-lined cap	Cool, 4°C, 0.008% $Na_2S_2O_3$ store in dark	7/40 days for waters[4] 14/40 days for soils[4]
Volatile organics	G, Teflon®-lined septum	Cool, 4°C, 0.008% Na_2SO_3 HCL to pH 2	14 days
EDB, DBCP	G, Teflon®-lined septum	Cool, 4°C, 0.008% $Na_2S_2O_3$[6]	28 days
Chlorinated hydrocarbons	G, Teflon®-lined cap	Cool, 4°C, store in dark	7/40 days for waters[4] 14/40 days for soils[4]
Total organic halogens (TOX)	G, Teflon®-lined cap	Cool, 4°C, H_2SO_4 to pH <2 store in dark	28 days[3]
Acid and base/neutral extractables	G, Teflon®-lined cap	Cool, 4°C, 0.008% $Na_2S_2O_3$[6] store in dark	7/40 days for waters[4] 14/40 days for soils[4]
TCLP and ZHE extraction	P,G	Cool, 4°C	14/NS/14 days for VOCs 14/7/40 days for organics 180/NS/180 days for metals 28/NS/28 days for mercury
Wisconsin GRO	G, Teflon®-lined septum	Cool, 4°C, 500 uL 50% HCl (Water) Cool, 4°C, 25 mLs MeOH (Soil)	4 days shipping/14 days analysis 4 days shipping/14 days analysis
Wisconsin DRO	G, Teflon®-lined septum	Cool, 4°C, 5 mLs 50% HCl (Water) Cool, 4°C (Soil)	4 days shipping/47 days analysis[10] 4 days shipping/47 days analysis
<u>Tissues</u>			
Organics, inorganics 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 = amber glass.
HCl = hydrochloric acid (metals grade).
HNO_3 = nitric acid (metals grade).
H_2SO_4 = sulfuric acid (metals grade).
NS = *none specified* by EPA.
MeOH = methanol.
Na_2SO_3 = sodium sulfite (ACS grade).
$Na_2S_2O_3$ = sodium thiosulfate (ACS grade).
P = polyethylene.
PCB = polychlorinated biphenyl.
NaOH = sodium hydroxide (ACS grade).
°C = degrees Celsius.
IC = ion chromatography.

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

[1] For nonvolatile organics, containers for soil and sediment samples are amber glass with Teflon®-lined caps and for volatiles, containers are amber glass with Teflon®-lined septum.
Soil sample containers for inorganics are amber glass jars with Teflon®-lined caps, polyethylene (P), or amber glass (G).

[2] 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 be 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.

[3] 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.

[4] 7/40 = 7 days until extraction; 40 days from extraction until analysis. 14/40 = 14 days until extraction; 40 days from extraction until analysis.

[5] 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). $Na_2S_2O_3$ is used only in the presence of residual chlorine.

[6] If residual chlorine is present, sodium thiosulfate is added to the sample first. It is not recommended to mix the two preservatives (and sample) together in an intermediate vessel.

[7] 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.

[8] 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.

[9] Temperature and pH must be measured on-site at the time of sample collection. Seven days is the maximum time for laboratory analysis of total alkalinity, calcium ion, and total solids.

[10] The holding time is the amount of time between receipt by the laboratory and addition of solvent to the sample. An exception will be allowed if samples arrive at the laboratory after 4:00 p.m. on a Friday. However, if the laboratory holds DRO samples over a weekend without adding the solvent to them, they must do so by 10:00 a.m. the following Monday. In no case may solvent be added past the 114 hours from the time of collection without flagging the data. It is not necessary for the laboratory to complete the extraction at the time of injection of the solvent.

Source: ESE.

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. Acetonitrile, hexane, HPLC grade methanol, and MTBE are stored in the GC/HPLC analysis area.
Inorganic acids	Stored in original containers in the ESE stockroom. Once taken from the stockroom to a department, the acids are stored in the department's cabinet or under a fumehood.
Organic acids	Stored in original containers in the ESE stockroom. Once taken from the stockroom to a department, the acids are stored in the department's cabinet or under a fumehood.
Caustics	Stored in original containers in the ESE stockroom. Once taken from the stockroom to a department, the caustic reagents are stored in the department's cabinet or under a fumehood. 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 area in each department. Liquids in quantities of one gallon or more, not stored in a cabinet, 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.
* Once removed from the storage room or while in use, reagent bottles are kept in safety carriers.

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 are 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 precleaned 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 7-3). Receipt of the sample containers is acknowledged by the field personnel by signing and dating the first line under the "Received By" on the Chain-of-Custody logsheet.
2. The formal Chain-of-Custody is signed by the Sample Custodian, or a designee, in the laboratory. In the field, the Field Team Leader or a designee is responsible to ensure that the Chain-of-Custody logsheet is maintained.
3. Copies of the Chain-of-Custody logsheets are maintained with project records.
4. Errors on all documents are corrected by striking one line though the error, then signing, and dating the corrections.
5. All documentation/logs are signed/initialed by appropriate personnel.

Due to the evidentiary nature of the samples collected, possession of the Chain-of-Custody must be traceable from the time the sample containers leave the laboratory to the time they enter the field. Field chain of custody actually begins at the laboratory. Sample kits, which refer to coolers, sample containers, preservatives, and trip blanks are requested from the kit preparation staff using the Container Order Form (Figure 7-1). This form is completed by the Laboratory Coordinator or Project Manager and accompanied by the labels (Figure 7-4) and any other relevant information. Shipping labels are provided in accordance with current corporate policy on sample kit handling.

The pre-preserved sample containers (The bottles are labeled with the appropriate preservatives and preservation codes (Figure 7-5).); trip blanks, if needed; and Chain-of-Custody logsheet are packed in coolers, sealed, and shipped to the field personnel by bonded carrier (i.e., UPS or Federal Express). All Container Order Forms are signed and dated upon completion by kit preparation staff. The number of coolers shipped to the field is documented on the Container Order Form and on the shipping receipts. An ESE Cooler Tracking Report (Figure 7-2) indicating the personnel who prepared the kits, cooler number(s), project name and number, and contents of each cooler is generated. The Cooler Tracking Report is kept on file by Sample Receiving personnel.

7.1.3 DOCUMENTATION

The records for laboratory sample custody include:

1. Laboratory Forms:

   Container Order Form (Figure 7-1),

   Cooler Tracking Report (Figure 7-2),

   Chain-of-Custody Logsheet (Figure 7-3),

   Sample Label (Figure 7-4),

   Standardized Sample Preservation Codes (Figure 7-5),

   Sample Custody Logbook (Figure 7-6),

Cold Room Sample Location Report (Figure 7-7),

Internal Chain-of-Custody (Figure 7-8),

Analysis Summary Form (Figure 7-9),

Internal Sample Arrival Notice (Figure 7-10),

VOA GC Sample Internal Chain-of-Custody (Figure 7-11), and

VOA GC/MS Sample Internal Chain-of-Custody (Figure 7-12).

2. Sample Extraction Log (Organic Laboratory/Extraction Logsheet, Figure 7-13, Metals Laboratory/Digestion Logsheet, Figure 7-14).

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Figure 7-1 Container Order Form

Environmental
Science &
Engineering, Inc.

Labels ____________

Container Order Form

Project Description: ______________________ Date: __/__/__

Submitted By: ______________________ Ship To: ______________________

Project Manager: ______________________

Must Have Containers By: ______________________

Ship: ❑ Std. ❑ Overnight ❑ 2nd Day

# Samples	Matrix	Parameter(s)	Containers			
			Type	Size	#	Preservatives

Special Instructions:

	Yes	No
Chain of Custody	❑	❑
Blue Ice	❑	❑
Return Labels	❑	❑
Sampling Instructions (type)	❑	❑

Prepared By: ______________________

Date Sent: __/__/__

Cooler #'s: ______________________

If there are any discrepancies, please contact ESE's Receiving Department at (800) 234-1239 immediately.

Figure 7-2 Cooler Tracking Report

Cooler #: Prepared by:

On Hand: No

Ship to:
Client:
Proj Num:
Address

City ST
ZIP

Date Sent
Proj. Mgr.

Comments
ROTATION

Special Instructions:

Yes	Chain of Custody (Y/N)
Yes	Custody Labels (Y/N)
Yes	Blue Ice (Y/N)
Yes	Return Labels (Y/N)
No	Sampling Instructions (Y/N)
Yes	MSDS Enclosed (Y/N)

SHIPPING

Yes	Std	-:
No	Overnight	-:
No	Second Day	-:
No	Three Day	-:

# Sxs	Matrix	Parameters	CONTAINERS: Type	Size	#	Preservatives

Figure 7-3 Chain-of-Custody Logsheet

ESE Environmental Science & Engineering, Inc.
8901 North Industrial Road -- Peoria, Illinois 61615
Telephone: (309) 692-4422 -- Fax: (309) 692-5232

FOR LAB USE ONLY
Project Number: ______
Due Date: ______

Chain of Custody Record

№ 6125

Company: ______
Address: ______

Phone #: (__)____-______ Fax #: (__)____-______
P.O. #: ______
Client Contact: ______
Project # / Location: ______

CONTROLLED DOCUMENT DO NOT DUPLICATE

Sample Type:
1. Water
2. Soil
3. Sludge
4. Oil
5. Tissue

Other : ______

Container Type:
P - Plastic
G - Glass
V - VOC

Preservative:
1. None
2. H2SO4
3. HNO3
4. NaOH

Sample I.D. (10 Characters ONLY)	Sample Type	Container Size	Container Type	Container No.	Sampling Date	Sampling Time	Preservative	Lab I.D.	Analyses	Comments

Relinquished By:	Date: -- -- Time: :	Received By:	Date: -- -- Time: :
Relinquished By:	Date: -- -- Time: :	Received For Lab By:	Date: -- -- Time: :

TURNAROUND TIME:
☐ RUSH:____ day turnaround
☐ ROUTINE

FOR LAB USE ONLY
Samples Received Chilled
☐ Yes
☐ No

SPECIAL INSTRUCTIONS:

Copies: White - Client Canary - Lab Receiving Pink - Lab File Goldenrod - Retained by Sampler

Figure 7-4 Sample Label

PROJ 591-5193 S402
FORT
IFFS01*1-C
SAMPLER DATE TIME
PH COND

PROJ 591-5193 S402
FORT
IFFS01*1-C
SAMPLER DATE TIME
PH COND

PROJ 591-5193 S402
FORT
IFFS01*1-EC
SAMPLER DATE TIME
PH COND

PROJ 591-5193 S402
FORT
IPFS01*1-Z
SAMPLER DATE TIME
PH COND

PROJ 591-5193 S402
FORT
IPFS01*1-S
SAMPLER DATE TIME
PH COND

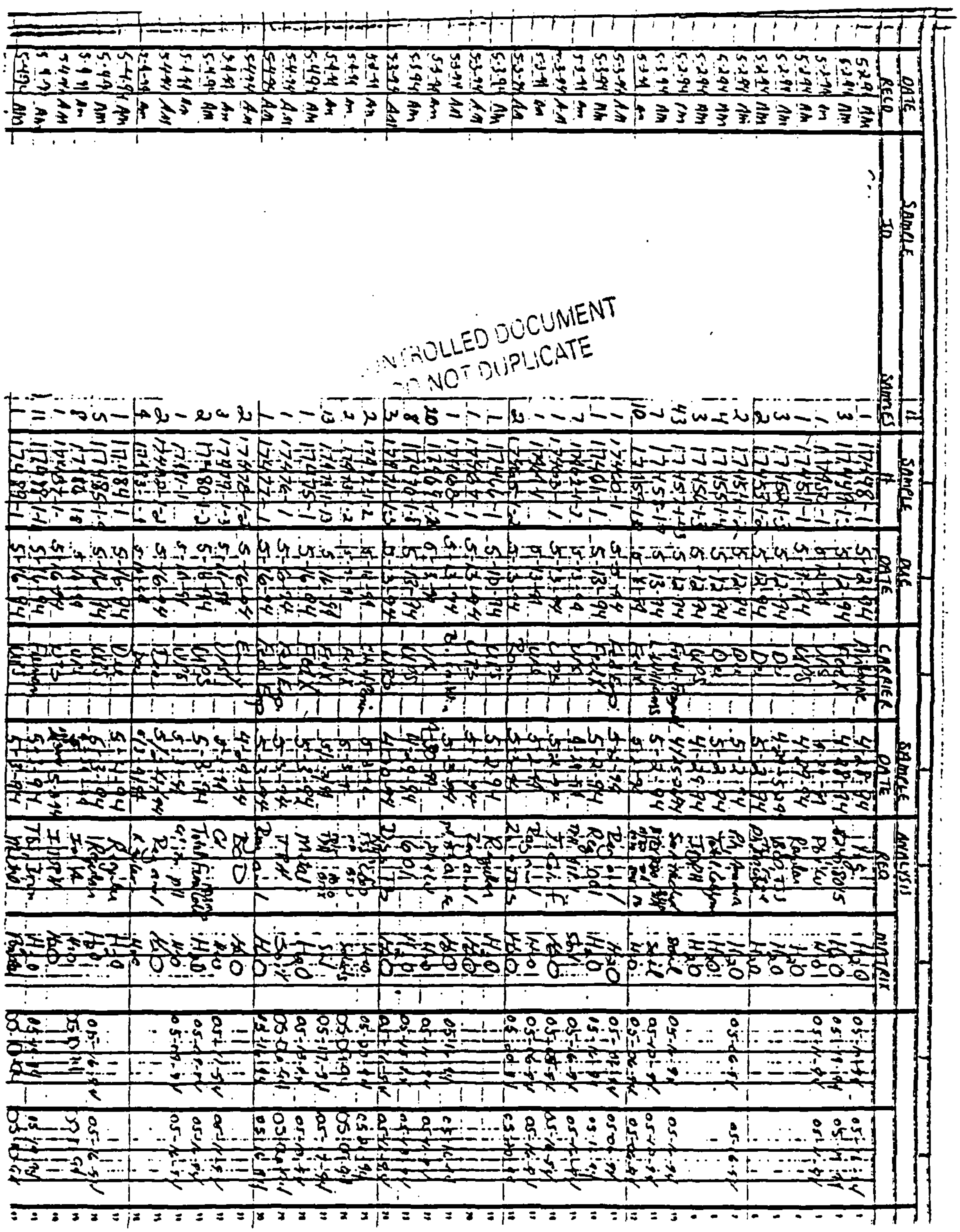
PROJ 591-5193 S402
FORT
IPFS01*1-N
SAMPLER DATE TIME
PH COND

Figure 7-5 Standardized Sample Preservation Codes

SAMPLE FRACTIONS AND PRESERVATIVES

FRACTION CODE	PARAMETER	CONTAINER SIZE/TYPE	PRESERVATION
C	Residues, Chloride, Sulfate, Fluoride, Bromide, Silica (dissolved), Specific Conductivity, Alkalinity, Acidity, Nitrate, Nitrite, Turbidity, BOD, Color, MBAS, Chromium (VI), Orthophosphate	1 L Plastic	4°C
S	COD, TOC, Kjeldahl Nitrogen, Ammonia, Total Phosphorus	1 L Plastic	4°C, H_2SO_4 to pH <2
O	Oil and Grease, TRPH	1 L Glass	4°C, H_2SO_4 pH <2
Z	Total Phenols	1 L Glass	4°C, H_2SO_4 to pH <2
N	Metals, Hardness	1 L Plastic	HNO_3 to pH <2
EC	Pesticides/PCBs	1 L Glass	4°C
MS	Acid and Base/Neutral Extractables, PNAs, Nitroaromatics	1 L Glass	4°C
V	Purgeable Compounds (Volatile Organic Compounds)	(2) 40-mL Glass Teflon-lined septum cap	4°C
VP	Purgeable Aromatics (BTEX)	(2) 40-mL Glass Teflon-lined	4°C HCl to pH <2
B	Cyanide, Total and Amenable to Chlorination (Free Cyanide)	1 L Plastic	4°C, NaOH to pH <2
X	TOX	(2) 250-mL Glass Teflon-lined septum cap	4°C, H_2SO_4 to pH<2
H	Sulfide	1 L Plastic	4°C, Zn Acetate, NaOH to pH >9
SS	All Solids (except VOCs)	250 mL Glass	4°C
SV	Volatile Solids	120 mL Glass	4°C

Figure 7-6 Sample Custody Logbook



DATE REC'D	SAMPLE ID	# SAMPLES	SAMPLE #	DUE DATE	CARRIER	SAMPLE DATE	ANALYSIS REQ	MATRIX

Figure 7-7 Cold Room Sample Location Report

[illegible] ROOM SAMPLE LOCATION REPORT FOR 05/24/94 01:32PM PAGE # 7

INITIALS	FLD.GRP.	FRAC	LOCATIONS	INITIALS	FLD.GRP.	FRAC	LOCATIONS	INITIALS	FLD.GRP.	FRAC	LOCATIONS
---	17657	V	C-02	---	[illegible]	SS	A-54	---	17658	SV	E-02
---	17659	EC	A-54	---	[illegible]	V/C	D-02	---	17659	V/EC	A-58
---	17660	C	A-54	---	[illegible]	D	A-54	---	17662	V/C	D-02
---	17662	V/EC	A-58	---	17663	[illegible]	A-54	---	17664	D	A-54
---	17664	C	A-54	---	17664	[illegible]	A-54	---	17665	SS	A-54
---	17666	C	A-54	---	17666	[illegible]	A-54	---	17667	N	A-54
---	17667	V	E-02	---	17668	N	A-54	---	17669	SS	A-54
---	17669	SV	E-02	---	17670	EC	A-54	---	17670	SS	A-54
---	17671	B	A-54	---	17671	N	A-54	---	17671	Z	A-54
---	17672	C	A-54	---	17673	B	A-01	---	17673	C	A-01
---	17673	N	A-01	---	17673	S	A-01	---	17673	X	A-01
---	17673	Z	A-01	---	17674	V	C-02	---	17675	SS	A-01
---	17675	SV	C-02 E-02	---	17676	D	A-01	---	17677	SV	C-01
---	17678	D	A-01	---	17679	SS	A-01	---	17680	B	A-01
---	17680	MS	A-01	---	17680	N	A-01	---	17680	O	A-01
---	17680	V	E-03	---	17680	Z	A-01	---	17681	SS	A-01
---	17682	C	A-01	---	17682	N	A-01	---	17683	N	A-01
---	17683	Y	A-01	---	17684	EC	A-01	---	17684	V	C-03
---	17685	EC	A-01	---	17685	V	C-03	---	17686	SV	E-03
---	17686	V	E-03	---	17687	SS	A-01	---	17688	SS	A-01
---	17688	SV	C-03	---	17689	C	A-01	---	17690	C	A-01
---	17691	C	A-01	---	17691	N	A-01	---	17692	C	A-01
---	17692	O	A-01	---	17693	V	C-03	---	17694	C	A-01
---	17694	S	A-01	---	17695	SV	C-03	---	17696	AIR	A-01
---	17695	SS	A-01	---	IPFS01	C	A-54	---	IPFS01	MS	A-54
---	IPFS01	N	A-54	---	IPFS01	S	A-54	---	IPFS01	Z	A-54

Figure 7-8 Internal Chain-of-Custody

INTERNAL CHAIN OF CUSTODY
Environmental Science & Engineering, Inc.

ESE Sample Number	Fraction Code Requested	Number of Bottles	Date	Time Removed	Initials	Date	Time Returned	Initials

Figure 7-9 Analysis Summary Form

Analysis Summary Form

Field Group: ____________ Seq. #: ______________ ESE Job #: ____________

Date Received: ______________________________ Turnaround Time: ______

Client Address: ______________________________ Engineering-Related? Y/N

______________________________ Fax Results? Y/N

______________________________ If yes, Fax #: __________

ATTN: ______________________________ Verbal Results? Y/N

DUE DATE: ______________________________ If yes, Phone #: __________

Section	Waters	Solids	Special Instructions
GC/MS Semivolatiles	______	______	______
GC/MS Volatiles	______	______	______
GC Extractibles	______	______	______
FID Extractibles	______	______	______
FID Volatiles	______	______	______
GC Volatiles	______	______	______
HPLC	______	______	______
Water Quality Short (Inorg. HT ≤ 14 days)	______		Cost: ______
Water Quality Short(Other)	______		______
Water Quality Long	______		______
Water Quality Long(Other)	______		______
Microbiology	______		______
Incineration Parameters	______		______
Metals -- GFAA	______	______	______
Metals -- ICP	______	______	______
Metals --Mercury	______	______	______

TCLP						
	Metals	______	Herb.	______	BNA Ext.	______
	Mercury	______	VOC	______	Pest Ext.	______
	Pest.	______	BNA	______	Herb Ext.	______

Total # of Pages in Project: ______________

Updated: 8-2-93

Environmental Science & Engineering, INC. 08/01/94 PAGE# 1-1 COPY#

*** SAMPLE ARRIVAL NOTICE FOR DEPARTMENT: TCLP MERCURY ***

PROJECT NUMBER
FIELD GROUP
ARRIVAL DATE: ____________
COLLECTION DATES: A=07/29/94

DUE DATE(S) : A=08/11/94

PROJECT NAME
PROJECT MANAGER
LAB COORDINATOR

MATRIX : SOLID DET. LIMIT SPEC'D : NONE SPECIFIED
SAMPLE FRACTIONS SS, SS

QC : STD

COMMENTS/QC : BDR 25 C

QC SUMMARY REQUIRED: [] YES [] no [] CLP Data Package [] FORM 1's Only

SDG #: ____________

SAMPLE COLLECTION DATE CODES: A
SAMPLE DUE DATE CODES: A

TAT: 1 3 7 30 days

SAMPLE DESCRIPTION/ID			EXTRACT GROUP	DET.LMT. CRITERIA	ANALYSIS DATE	ANALYST INITIAL	1 SOLIDWASTE
MERCURY, TCLP	,MG/L	71900*PTCLP	TCLP		______	______	______

______ =OPEN L=IN LAB E=EXTRACTED N=NOT REQUESTED D=DONE S=SCHEDULED
* = STORET NOT ON DEPT AVAIL NUMS

DO NOT DUPLICATE
CONTROLLED DOCUMENT

Other Depts Using Samples:	1803
	TCLP ICAP METALS
ONLY 1 BOTTLE AVAILABLE	TCLP MERCURY
[] YES [] No	WATER QUALITY SHORT
	WATER QUALITY SHORT --
	WATER QUALITY LONG
	TCLP SEMIVOLATILES
	TCLP VOLATILES

Correct Units Reported? <> Yes <> NO
Analysis Date in CLASS: <> Yes <> NO
Correct Analyst's ID: <> Yes <> NO
Reviewer's Initials: ____________

Method Checklist		
METALS	ORGANICS	CLP
SW846 []	SW846 []	3/88 []
	600s []	
200s []	500s []	3/90 []

Figure 7-10 Internal Sample Arrival Notice

Figure 7-11 VOA GC Sample Internal Chain-of-Custody

VOA — FIO — ECD — NP — EC — · LOGIN

PIN#	CLIENT	PARA	BATC	HOLD/ EXT.	ANAL	DUE	T.I.	DONE

PAGE____

Figure 7-12 VOA GC/MS Sample Internal Chain-of-Custody

GC/MS SAMPLE LOG VOLATILES

FIELD GROUP	SEQ #	S/W	CLIENT	ANAL	DATE DUE	DATE REPT	CLASS BATCH	# DAYS

CLLED DOCUMENT
NOT DUPLICATE

Figure 7-13 Organic Laboratory/Extraction Logsheet

ESE Environmental Science & Engineering, Inc.

GC Sample Prep Log

Page ______

Date Extracted: ___/___/___ Extract Solvent: ____________ Date Concentrated: ___/___/___ Extraction No.: ____________

Extractors: ____________ Cleanup: ____________ Final Solvent: ____________ Book No.: ____________

	Client	Method No.	Sample ID	Initial/ Final pH	Sample Vol. (l)/ Weight (gm)	Surrogate Added ID no.	Surrogate Added Vol. (ml)	Spike Added ID no.	Spike Added Vol. (ml)	Final Vol. (ml)	Comments
1											
2											
3											
4											
5											
6											
7											
8											
9											
10											
11											
12											
13											
14											
15											
16											
17											
18											
19											
20											
21											
22											
23											
24											

Comments ____

Figure 7-14 Metals Laboratory/Digestion Logsheet

METALS SAMPLES PREPARATION

PREP BATCH#__________

MATRIX:	Start Date: Ini.	METHOD:
	End Date: Ini.	

For:	Rack loc.	pH <2	Sample Wt./Vol.	Vessel Wt. B	Vessel Wt. A	Final Vol.	Homogen. Y/N	Comments:
QC SAMP.							SPK. SOLUTIONS	
SPM1		NA						
SPM2		NA						
RP		NA						
MB		NA						
LCS		NA						

B = vessel, sample & acid before dig.
A = vessel, sample & acid after dig.

%Loss = B-A/15.2 X 100
15.2 = 10ml HNO3 + 0.5g sample

Page #______.

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

7.2 FIELD CUSTODY PROCEDURE

To establish the documentation necessary to trace sample possession from the time of collection, a Chain-of-Custody record is completed and accompanies every sample. This record becomes especially important if the sample is to be introduced as evidence in court litigation. The record contains the following minimum information: sample description and matrix, analyses requested, signature of collector, date and time of collection, signature of persons involved in the chain of possession, and comments such as suspected hazards or visible/suspected physical characteristics of the sample.

In collecting samples for evidence, only the number of samples which provides a good representation of the media being sampled are taken. To the extent possible, the quantity and types of samples and sample locations are determined prior to the actual field work. As few people as possible handle the samples. The samples are under the direct control of the field sampler for that project.

The field samplers are personally responsible for the care and custody of the samples collected until they are transferred or dispatched properly.

Sample labels are completed for each sample using waterproof ink, unless prohibited by weather or other special conditions. For example, a logbook notation could explain that a pencil was used to fill out the sample label because a ballpoint pen would not function in freezing weather. Labels are affixed to sample containers prior to the time of sampling. The labels are filled out at the time of sampling.

The field supervisor determines whether proper custody procedures were followed during the field work and decides if additional samples are required.

If at any time the samples are to leave the immediate and direct control of the field sampler prior to delivery to ESE, cooler seals are used to detect unauthorized tampering. Cooler seals are gummed paper or similar material. The paper seal includes the following minimum information: Collector's name, date, and time of sampling, identifying number or reference.

The cooler seal should be attached in such a way that is necessary to break it in order to open the shipping container. Seals are affixed to the containers before the samples leave the custody of sampling personnel unless the samples are transferred directly from the field sampler to the authorized Sample Custodian of ESE.

7.3 TRANSFER OF CUSTODY AND SHIPMENT - FIELD TO LABORATORY

Samples are delivered to ESE for analysis as soon as practical - usually within one or two days after sampling. The samples are accompanied by the Chain-of-Custody completed by the field sampler at the time of collection and delivered to the Sample Custodian or the designee.

When transferring the possession of samples, the individuals relinquishing and receiving shall sign, date, and note the time on the Chain-of-Custody. This record documents sample custody transfer from the sampler, to the laboratory and subsequently, sample storage. Each individual who signs the Chain-of-Custody has a responsibility to ensure that all information added to the Chain-of -Custody is complete and accurate.

Samples are packaged properly for shipment (including custody seals) and dispatched to ESE for analysis, with a separate Chain-of-Custody accompanying each shipment (each ice chest). The method of shipment, courier name(s), and other pertinent information is entered into the "Comments" section of the Chain-of-Custody.

7.4 LABORATORY CUSTODY

Sample chests (packages/coolers) are transported to the laboratory. The Sample Custodian, or designee, then signs the Chain-of-Custody indicating receipt of the samples by the laboratory. The Sample Custodian records the samples as having been received by the laboratory in the Sample Custody Logbook (Figure 7-6). The information recorded includes sample receipt date, morning or afternoon designation, sample identification (client), the number of samples received, unique laboratory identification number, the analysis due date, the sample carrier (e.g. UPS, Federal Express), the sampling date, analyses requested, and the sample matrix (matrices).

The samples are checked in by the Sample Custodian for proper preservation (e.g. pH, temperature), integrity (e.g., leaking, broken bottles, tainted custody seals), and proper, complete sample documentation and identification. Sample chests or coolers that are not within the 4 $\pm$ 2 degrees Celsius (°C) requirement are reported immediately to the Project Manager to determine if resampling will be required. All samples contained in the shipment are compared to the Chain-of-Custody to ensure that all samples designated on the custody record have been received. The Sample Custodian notes on the Chain-of-Custody any special remarks concerning the shipment. Any marks or notes made on the Chain-of-Custody document by the Sample Custodian are clearly distinguished from original field notations. The Sample Custodian reviews the integrity of all sample fraction containers and checks the accuracy and clarity of all documentation received. The Sample Custodian audits daily the first shipment of representative samples of all fractions requiring field preservation to ensure that they have been properly preserved. The audit is recorded in the Receiving pH logbook. The Sample Custodian preserves unpreserved fractions or adds additional preservative, if needed, upon receipt. Deficiencies in sample preservation, additional preservative added, and all other inadequacies are recorded on the Chain-of-Custody and reported to the Project Manager. The Project Manager, upon consultation with the client/field team, decides if resampling is required. The original Chain-of-Custody is sent back to the client with the final report. A copy of the Chain-of-Custody is kept in the internal project file with a copy of the final report.

The accepted samples are logged into the ESE laboratory LIMS (Laboratory Information Management System), CLASS™ (Section 7.5) using the unique laboratory sample identifications, which includes the ESE project identification number and sample ID provided by the sampler on the Chain-of-Custody. The sample collection date and receipt date are recorded and are used for monitoring holding time and progress of the project throughout the laboratory. The requested analyses are assigned to the individual samples and sample arrival notices (Figure 7-10), which are used for internal project tracking, are generated. The arrival notices are distributed to the appropriate laboratory sections by the Project Coordinator to notify the analysts of the arrival of the samples, identification and number of samples, required analyses, due dates, and specific QC requirements. Any special instructions or notes listed on the Chain-of-Custody will be mentioned on the arrival notice. To facilitate intralaboratory communication, the Sample Custodian who logged in the samples and the Project Manager for the project are recorded on the arrival notice. The arrival notices with the attached preparation logbook pages for samples requiring preparation before analyses, such as organic extractions and metals digestions, are forwarded to the appropriate analytical instrument section after the preparation has been completed. Any problems or observations noted during the preparation process are recorded on the arrival notice and entered into CLASS™. An Analysis Summary Form (Figure 7-9) is also created and filed in the project folder to track which departments received samples. Upon completion of the analyses required for the samples, the arrival notices are returned to the project folder and their completion noted on the Analysis Summary Form. The final report is then generated by an Administrative Assistant. Tracking the samples through the laboratory is done by the Work-in-Progress report which is distributed to project management, operations management, department management, and QA/QC. The Work-in-Progress report, created by an Administrative Assistant, is a daily register of all samples within the laboratory, listing the client name, project identification number, number of samples for that project, date received, departments receiving the samples, due date, and status (for example, a rush status could be listed).

Samples are placed in appropriate storage areas in the laboratory depending on storage requirements. The majority of the samples are stored in the main coldroom, with the exception of volatile samples, with the temperature maintained at 4 ± 2°C. The samples in this storage area are arranged by field group. The main coldroom is kept locked after normal working hours. Volatile samples are refrigerated at 4 ± 2°C and stored in the GC and GC/MS Volatiles area. An Internal Chain-of-Custody (Figure 7-8) is maintained for the coldroom as well as the GC volatiles refrigeration unit (Figure 7-11) and GC/MS volatiles refrigeration unit (Figure 7-12). A Cold Room Sample Location Report (Figure 7-7) is generated weekly to facilitate sample retrieval. Sample storage areas are used only for sample storage. Samples remain in storage for one month after receipt into the laboratory unless otherwise directed by the client. Sample extracts remain in storage for one month after analyses unless otherwise directed by the client.

During normal work hours, there are always laboratory staff present in the ESE Peoria Laboratory. Entry to the building for visitors is available through the front door of the main building or the ESE Peoria Laboratory receiving area located at the north side of the main building. A receptionist is present at the front door to greet visitors. Visitors must sign a visitor's register and are escorted through the building by ESE personnel. The building is continuously locked and is secured with a Security Link® Alarm System after normal working hours.

When it is necessary to use another laboratory for sample analysis, the Project Manager is responsible for arrangements with the second laboratory. The samples are only subcontracted to a state or federal government agency, or client-approved laboratory. The Chain-of-Custody accompanies samples transferred to another laboratory and includes the following information: collection data and time, field ID, laboratory ID, date of sample preparation, and requested analyses.

The samples are kept at 4 ± 2°C prior to and during shipment. A Chain-of-Custody indicating samples and fractions sent accompany the samples to the subcontractor. The subcontractor signs and dates the Chain-of-Custody upon receipt of the samples. A copy of the signed Chain-of-Custody is returned to ESE and placed in the project file.

7.5 LABORATORY INFORMATION MANAGEMENT SYSTEM (LIMS)

CLASS™ is an automated, in-house-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 fileserver using Novell Netware version 3.12, and contains 1.6 gigabytes of storage. In Peoria, the network is connected to more than forty personal computers, and via the Wide Area Network, connected to all other ESE laboratories and engineering facilities. CLASS™ is managed by the Laboratory Information Services Department within the Peoria Laboratory, with support from the ESE Gainesville Laboratory Information Services Department. 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 Revelation Technologies). 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. The Project Manager provides information on the number of samples, site IDs, parameters to be analyzed, and estimated collection dates prior to sampling, if applicable. This information is entered into CLASS™ and used to produce sample labels. 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 Chain-of-Custody. Samples accompanied by the Chain-of-Custody are sent to the laboratory where they are checked, processed, and stored by the Sample Custodian. The samples, along with the date of collection and site identification, are logged into CLASS™ by the Sample Custodian. Chain-of-Custody forms are placed in the project file and maintained by the Laboratory Coordinator.

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 sample arrival notice 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 regularly produced and distributed to each laboratory department.

CLASS™ uses a batch method for analyzing, checking QC, and calculating final results of samples. Prior to analyzing a sample batch, the analyst designates a specified group of samples in the computer and the sample-parameter status is updated. 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. Usually, instrument calibrations are entered first and include several QC checks by CLASS™. The linear (or quadratic) 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. The batch along with the raw data are reviewed and signed by the Department Manager or a designated reviewer.

The Department Manager or designated reviewer processes the batch in the computer to verify QC and to update the sample records and final calculated concentrations. Once a batch has been finalized by the Department Manager or reviewer, the batch is locked and data cannot be changed. The final report is then generated and reviewed by the ESE Project Manager before it is sent to the client. If batch edits are required, the LIMS Manager is notified and definalizes the batch. Changes and refinalization are done by the appropriate Department Manager. The original and revised batch reports are found in the batch folder, along with documentation concerning the reason for the batch definalization.

Each employee is assigned an individual access code for entry into CLASS™. All personnel with an access code may retrieve information from the system. Access rights are assigned on an individual basis. Laboratory personnel are not allowed to update sample records without authorization from the LIMS Manager. Only personnel with appropriate access codes and LIMS Manager approval may edit laboratory data.

The batch folders, with all supporting documentation (such as organic extraction log pages (Figure 7-13) and metals digestion log pages (Figure 7-14)), 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, Project Coordinators, Project Managers, Department Managers, or QA/QC personnel. A Document Control Logbook (Figure 12-3) is used to track folders that have been checked out. Batch folders and project files are kept a minimum of ten years.

Laboratory personnel 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. CLASS™ enables a Laboratory Coordinator or Administrative Assistant to:

1. Produce a variety of summary reports of analytical data,
2. Produce sample summary reports,
3. Calculate statistics such as mean, maximum, minimum, and standard deviation,
4. Summarize QC in various formats, and
5. 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 regular hardcopy 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 supports 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 name 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 QA staff checks data packages quarterly, including computer printouts, to verify that CLASS™ data match raw data from the laboratory.

The database is backed up daily except Saturday using high-density storage media. The tapes are stored in the Information Services air-conditioned locked office.

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 operating procedures are required for all departments and development of the documents are ultimately the responsibility of the Department Managers. The methods cited in these documents are the methods normally used. Any deviation from the standard method is 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 are validated as described in Section 8.2.

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. Method validation is warranted when 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. The responsible Department Manager or analyst must establish a thorough method validation so that the selected method measures the reported parameter with the necessary precision, accuracy, and detection limit, without severe interference by other constituents in the sample. If required, nonstandard methods and validation documentation will be submitted to state or government agencies (i.e. IEPA, USACE, etc.) and clients for review and approval prior to use on samples for analyses.

The requirements for method validation include the performance of an Initial Demonstration of Capability and Method Detection Limit Study. The following subsections constitute the minimum requirements for initial establishment of the accuracy, precision, and detection limits of nonstandard methods.

8.2.1 INITIAL DEMONSTRATION OF CAPABILITY

For each parameter of interest, a minimum of four replicate spike samples are prepared from laboratory blank water at one appropriate analyte concentration. Spiked samples are analyzed according to the method. An unspiked "standard" matrix blank or unspiked laboratory blank water is analyzed. The spiking concentration is selected such that the final extract or aliquot is analyzed in the midrange of the calibration curve.

The Initial Demonstration of Capability protocol is summarized below:

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

1. Calculate the found concentration for each spiked sample as follows:

   R = measured concentration = measured concentration in spiked sample minus the measured concentration in unspiked (blank) sample.

2. Calculate the Percent Recovery (P) for each spiked sample as follows:

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

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

3. Calculate the Average Percent Recovery (P_{ave}), Standard Deviation of the percent recoveries (S_r), and Percent Relative Standard Deviation of the percent recoveries (RS_r) of the spiked samples as follows:

$$P_{ave} = \frac{P_1 + P_2 + P_3}{3}$$

where: P_1, P_2, and P_3 = percent recovery of the three spiked samples

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

where: S_r = standard deviation of P_{ave}

$$RS_r = \frac{S_r}{P} x\ 100$$

where: n = number of recovery values, and
RS_r = relative standard deviation of P.

<u>Precision</u> The minimum requirements for establishment of precision for methods are as follows:

1. Calculate the Relative Percent Difference (RPD) between each pair of replicate spiked samples.

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

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

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

2. Calculate the average RPD for the spiked samples.

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

8.2.2 METHOD 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 are used. For each parameter of interest, a minimum of seven replicate spike samples are prepared from laboratory blank water at one appropriate analyte concentration. Spiked samples are analyzed according to the method. An unspiked "standard" matrix blank or unspiked laboratory blank water are analyzed. The spiking concentration is selected such that the concentration is approximately one to ten times the estimated or method detection limit for the parameter.

The reported detection limit for a method is subject to the judgment of the analyst and the Department Manager and takes 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 determination results (Initial Demonstration of Capability and Method Detection Limit studies) are recorded and submitted to the Department Manager and Laboratory QA/QC Coordinator prior to the initiation of analysis. Before analysis begins, the Department Manager assures that the method meets the performance criteria required by the project.

Once the method is validated, the 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 glassware is drained of solvents and rinsed with tap water when soils or other residues are still remaining, before it is washed.

All laboratory glassware (i.e., volumetric flasks, separatory funnels, beakers, graduated cylinders, etc.) is 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.

8.4 LABORATORY METHOD MODIFICATIONS

Laboratory method modifications are done either to improve the method efficiency 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). These compounds are denoted and their QA targets found in Section 5.0. Initial Demonstrations of Capability and Method Detection Limit studies were performed for the compounds.

8.5 REAGENT STORAGE

The procedures for storing reagents in the laboratory are presented in Section 6.5. All reagents are marked with initials, date received, and date opened.

Table 8-1. Glassware Cleaning Procedures

Analysis/Parameter	Cleaning Protocol*
Extractable Organics	1,2,3,4,5,8,9
Purgeable Organics (Volatiles)	5,4,8
Trace Metals	1,2,3,5,6
Nutrients, Minerals, Demands, Cyanide, Phenols	1,2,3,5
Gravimetric, e.g. Residues, Oil and Grease	1,2,3,5,8
Phosphorus, All forms	1,2,3,7,5

Note: HCl = Hydrochloric acid
HNO_3 = Nitric acid

*Cleaning Procedures

1. Remove all labels using sponge or brush.
2. Wash with hot soapy water (use Liquinox soap only) using brushes to scrub inside of glassware, stopcocks, and other small pieces if possible.
3. Rinse three times with tap water.
4. Rinse three times with histological grade methanol.
5. Rinse three times with deionized water.
6. Acid rinse with dilute HNO_3 and then with tap water.
7. Acid rinse with 1:1 HCl and then with tap water.
8. Bake at 180°C for 1 hour or until dry.*
9. Rinse with appropriate extraction solvent prior to use.

* Class A volumetric glassware should not be baked.

Source: ESE.

8.6 LABORATORY WASTE DISPOSAL

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

8.6.1 LIQUID WASTES

In general, no chemical wastes are disposed in the sinks without contacting the Department Manager or Hazardous Waste Coordinator (HWC). Only certain dilute acid wastes are disposed in the sinks.

8.6.1.1 Acid Wastes

All acid waste (not containing heavy metal concentrations to be considered a "regulated waste") generated by the Atomic Spectroscopy and Water Quality Department as digestates and instrument waste are disposed in the designated Acid Waste plastic drum located in the Metals Digestion area and the digestion tubes discarded. All TCLP extracts are disposed in the designated Acid Waste containers located in the Metals Digestion and Water Quality areas.

8.6.1.2 Disposal of Standards and Solutions

As standards and solutions are made, the solvent, constituents, date prepared, expiration date, reference number, and initials of preparer must be put on the container. This information must be on the container before it is disposed by the HWC. Standards containing any amount of organic solvent are not poured down the sink. Aqueous standards containing organic or inorganic (metals, etc.) compounds are either disposed in the appropriate waste drum, or picked up by the HWC.

8.6.1.3 Disposal of Solvent Wastes

All waste solvents are disposed in approved solvent waste containers located throughout the departments in the laboratory. Solvents are segregated according to the designated chemical types and placed only in the appropriate waste container. The waste containers are emptied on a regular basis by the HWC. If the containers become full before then, the HWC will be called so the containers can be emptied.

Solvents will be segregated as follows:

Pentane/Fuel:	Fuel, oil, pentane
Freon:	1,1,2-Trichlorotrifluoroethane only
Chlorinated:	Methylene chloride, chloroform
Non-Chlorinated Flammable:	Acetone, benzene, cyclohexane, ethyl ether, hexane, isopropanol, petroleum ether, toluene, xylenes, MTBE, carbon disulfide
HPLC:	Acetonitrile, isopropanol, methanol, ethanol, water, carbamate analysis waste (OPA, NaOH, sodium borate, 2-mercaptoethanol)
Mixed Chlorinated Flammable:	Mix of methylene chloride, acetone, hexane, ethyl ether

Isopropanol may be disposed in either the Non-Chlorinated Flammable or HPLC container. Freon-112 is disposed only in designated waste containers in the Water Quality department, and not combined with other chlorinated wastes. Glass solvent containers are not accepted for solvent waste. Solvents are segregated as described and placed in designated waste containers.

8.6.1.4 Disposal of Extracted Water Samples

Water samples which have been solvent extracted are neutralized and disposed in the designated Sample Preparation department waste sink.

8.6.1.5 Disposal of Inorganic Wastewater

Samples and waste generated from the analysis of inorganic parameters such as phenols and cyanide are disposed in the designated waste containers in the Water Quality department.

8.6.2 SOLID WASTES

8.6.2.1 Solvent Saturated Soil and Solids

Non-hazardous waste such as packing materials, tape, or plastic wrap are disposed in trash receptacles. Cardboard, paper, aluminum, glass (not for laboratory use), or polyethylene plastic is recycled and is taken to the Sample Receiving department. Broken laboratory glass is placed in the repair box or disposed in the designated broken glass containers. Solvent saturated solids and filters, such as sodium sulfate or soil saturated with methylene chloride, are never disposed in trash receptacles. These solid wastes are placed in solid waste cans located in each department. These waste cans have lids to prevent fumes from entering the laboratory air. Full containers are collected by the HWC on a regular basis. The soil waste is taken to the HazWaste Area and placed in the Soil Trough. The contents of the Soil Trough will be allowed to air dry and disposed in the dumpster by the HWC.

8.6.2.2 Disposal of Expired or Contaminated Chemicals

Commercial chemicals, solvents, and standards that are out of date or contaminated are left in their original containers. The container is labeled, including the date and initials, prior to disposal. The HWC is then called to pick up the material.

8.6.2.3 Disposal of Autosample Vials Containing Extracts

All autosample vials are collected in the designated containers in each department. The containers are emptied on a regular basis by the HWC. If the container becomes full, the HWC is called.

8.6.2.4 Disposal of Additional Hazardous Material

The contents are clearly marked on the container or on an accompanying analysis report. The containers are dated and initialed. The HWC is contacted for pickup and disposal.

8.6.3 UNKNOWN WASTES

If an unmarked container or unknown waste is found, it is brought to the attention of the Department Manager or HWC. Unknowns are not allowed to accumulate. Unknowns are identified prior to disposal.

8.6.4 SAMPLE WASTES

After the completion of a project, an Administrative Assistant generates two final reports. The Project Manager sends the original to the client and indicates on the duplicate report whether to "Dispose", "Hold" and "Dispose On", or classify as "Hazardous" the samples for that project. The report is then distributed to the Sample Receiving Technician (SRT) who coordinates sample disposal. If the samples cannot be disposed due to recommendations or requirements and the date is after the six week sample shelf-life, the Project Manager marks "Hold" and a "Dispose On" date on the report to indicate which samples are to be retained. If there is no indication the samples are a regulated waste or pose a hazard to individuals working with the samples, the Project Manager marks "Dispose" on the report, indicating which samples are to be disposed. Ultimately, the Project Manager signs and dates the report authorizing disposal. If the samples are not regulated as a waste, but the samples pose a hazard to individuals handling them (odorous, etc.), the Project Manager indicates the concern on the report. The Project Manager then signs and dates the report to authorize disposal. If the samples are considered "Hazardous Waste", the Project Manager indicates and highlights "Hazardous" on the report. The Project Manager signs and dates the report authorizing disposal. Once the report is received by the SRT, the appropriate samples are removed the storage unit. If the samples are not "Hazardous" and not a regulated waste per the specified analysis, the SRT bulks the samples together for disposal.

8.6.4.1 Disposal of Water Samples

All water samples are neutralized and disposed by the SRT. The empty containers are recycled. All labels are removed prior to disposal. Appropriate protective equipment is worn when handling samples and containers.

8.6.4.2 Disposal of Soil/Solid Samples

All soil/solid samples are disposed by emptying the contents in the Soil Waste Drum in Sample Receiving. All labels are removed prior to disposal. Appropriate protective equipment is worn when handling samples and containers.

8.6.4.3 Disposal of Hazardous Samples

If the samples are regulated or classified as "Hazardous", the samples are either disposed or lab-packed by the HWC, depending on the classification. The HWC stores the samples in the designated sample waste area. The analysis report is kept in the HWC's waste files until the next hazardous waste pickup. During the storage time, the HWC combines all compatible samples to achieve the smallest overall volume.

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. Since some parts of the instrument are utilized more than others, replacement for these parts is required more frequently. These wearable or expendable parts are monitored during analysis for optimum performance and kept in supply in the event of instrument failure. Major instruments in the laboratory are covered by service contracts or agreements provided by various vendors.

10.1 DOCUMENTATION

All maintenance performed on the instruments is 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 the maintenance logbook. Receipts from the routine maintenance performed by the service representative are filed in the laboratory. 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 is made to analyze samples within holding times by alternate means. If ESE Peoria's additional instrumentation is insufficient to handle the affected samples, efforts are made to secure the same or equivalent analyses by an appropriately certified or validated laboratory. After contact with an alternate laboratory, the Project Manager is advised of any required changes in methodology or sample location; the Project Manager then notifies the appropriate state/government agency and the client of project modifications. Procedures concerning laboratory custody of samples is found in Section 7.4.

Table 10-1. Preventive Maintenance

Instrument	Activity	Frequency
Gel-Permeation	Replace sample/air syringe	As needed
	Check solvent flow	Daily
	Clean injectors	As needed
	Clean/replace guard column frits	As needed
	Change GPC columns	As needed
	Clean detector	As needed
Gas Chromatographs	Change septums	As needed
	Check carrier gas	Daily
	Change carrier gas	As needed (when pressure falls below 500 psi)
	Cut off edge of a capillary column	As needed
	Replace oxygen traps used in the gas lines	As needed
	Clean detectors	As needed
	Replenish detectors	As needed
	Clean detectors	Daily or as needed
	Check system for gas leaks	As needed
	Clean injection ports	Weekly or as needed
High Performance Liquid Chromatographs	Check piston seals	Weekly, replace as needed
	Check, replace or rebuild the the check valves	Weekly (replace/rebuild as needed)
	Clean detector flow cell	As needed
	Check pumps	Daily
	Replace guard column frits	As needed
	Clean detectors	As needed
	Degassed and leak checked	Daily
	System/air pressure	Daily
	Auto-injector syringes	Daily
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 liners	As needed
	Change pump oil	As needed
	Check flow level	As needed
	Routine maintenance performed by the manufacturer	Annually

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

Instrument	Activity	Frequency
Atomic Absorption Spectrophotometers (Furnace and Cold Vapor)	Clean furnace windows	Daily
	Check plumbing connections	Daily
	Change graphite tubes	As needed
	Clean sample cells	Daily
	Check gases	Daily
	Check optics and routine maintenance by the manufacturer	Annually (on contract)
	Change graphite contact rings	As needed
Inductively Coupled Plasma (ICAP)	Routine maintenance performed by the manufacturer	Annually (on contract)
	Check and clean the torch, nebulizer, and O rings	As needed
	Check tubing	As needed
Cold Vapor Analyzer	Clean adsorption cell	Daily
	Clean gas/liquid separator	Daily
	Replace pump tubing	Weekly
	Change drying column	Weekly
Autoanalyzers	Clean or replace tubing	As needed
	Check tubing	Daily
	Check and clean optics	As needed
	Clean flow cell	As needed
	Replace the lamp	As needed
Colorimeter/ Turbidimeters	Check optics	Daily
	Check light source	As needed
Spectrophotometer	Calibrate wavelength	Semiannually
	Replace lamps	As needed
	Replace phototubes	As needed
TOX Analyzer	Clean electrodes	Daily
	Replace all solutions	Daily
	Clean absorber module and the furnace unit	As needed
	Clean sampler boat	As needed
	Check gases and tubing	Daily
	Rebuild agar bridge	As needed
TOC Analyzer	Check gases and tubing	Daily
	Change pump tubes	As needed
	Flush system	After each use

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

Instrument	Activity	Frequency
Ion-analyzers/Conductivity	Check probe	Daily
	Change probe solution	As needed
Ion Chromatograph	Check system for leaks	Weekly
	Check line pressure and piston seals	Weekly
	Clean cell electrodes	Monthly
	Clean injection loops	As needed
	Change columns	As needed
	Replace tubing in the sample path	As needed
Turbidimeter	Clean the instrument	Daily
Analytical Balances	Clean the balance	Daily
	Check alignment and balance	Daily
	Routine maintenance and calibration performed by the manufacturer	Semiannually
Ovens: TS, TSS, TDS	Check temperature	Daily
	Calibrate thermometers	Annually
Refrigerators/Freezers	Check temperature	Daily
	Calibrate thermometers	Annually
BOD Incubator	Check temperature	Daily
	Calibrate thermometers	Annually

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

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) when the instrument detector has been subject to major maintenance, or (3) when the instrument fails the calibration QC checks.

All analytical instruments are calibrated with a series of standards. The series of standard solutions is prepared from stock standards. These standards are either purchased from various vendors in premixed solutions or prepared directly from stock compounds. The preparation of all standard solutions is documented in logbooks. All stock standards are inscribed with date received, date opened, date prepared (laboratory), and expiration date. The standards are stored in designated areas and checked for expiration on a regular schedule. Specific calibration requirements for major classes of analytical procedures are described in the following sections.

9.1 STANDARD RECEIPT AND TRACEABILITY

A standard is a solution of an analyte of interest with verifiable accuracy which is used to evaluate that constituent in a sample. 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 target compound must be verified and the accuracy requirements for its measurement available. The manufacturer ensures this through certification and traceability statements, which are kept on file in the laboratory. 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), supplied by the manufacturer.

Upon receipt, the standard is cross referenced with its purchase order to confirm that what was received is what was ordered. The chemical is checked with the purchase order and is placed on a table in a central area that is checked daily by the Department Manager. The standard receipt date is noted on each standard. All standards are stored in designated areas for each department.

9.2 STANDARD SOURCES AND PREPARATION

All standards 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. All initial standard preparations are recorded in appropriate logbooks. The information recorded is the standard prepared, the source and concentration of the standard, the expiration date of the neat chemical used to prepare the standard, the standard lot number, date prepared, and initials of the preparer. The protocol for standard sources and preparation is located in Table 9-1. All standards should not exceed the storage (use) life of both the stock and working solutions. Each working solution and stock solution is labeled with date prepared, expiration date, initials, concentration used, and reference number of the appropriate standard preparation logbook.

Secondary dilutions made from stock standards are also recorded in a logbook. The lot number of the stock standard used and the notebook number and/or page number are also 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

All laboratory instrumentation is listed in Table 9-2. Since calibration criteria is required for analytical operations, each of these instruments are calibrated in a manner consistent with EPA calibration protocols and/or ESE SOPs. Calibration is documented in an analysis logbook.

Table 9-1 Standard Sources and Preparation

Instrument Group	Standard Source(s)	Condition Received	Storage	Standard Preparation	Lab Stock Storage	Preparation Frequency
ICAP GFAA CVAA FLAA	Various	1,000 or 10,000 ppm soln.	RT	Intermediate and/or Working Stock	RT	> 1 ppm Monthly < 1 ppm Daily (GFAA)
Autoanalyzer	Various	Neat and/or Solution	RT	Primary Intermediate Working	RT RT RT	Monthly Biweekly Biweekly
IR	Various	Neat (oil) and/or Prepared	RT	Combined Primary Intermediate Working	RT RT RT	Quarterly Monthly Monthly
Inorganics	Various	Neat and/or Prepared	RT	Primary Working	RT RT	Quarterly Daily
GC (non-VOA)	Various	Neat, Mix, and/or Prepared	Freezer and/or Refrig.	Primary Intermediate Working	Frez/Ref Refrig. Refrig.	Semiannual Method Specific Monthly
GC (VOA)	Various (Ultra- Scientific)	Neat Mix Solution Solution	Freezer Freezer Freezer	Mixed Primary Intermediate Working Working Mixed Primary Intermediate Working	Freezer Freezer Freezer Freezer Freezer Freezer Freezer	Monthly Weekly Daily Weekly Monthly Weekly Daily
LC	Various	Neat Mix Solution	Frez/Ref Frez/Ref	Primary Intermediate Working Primary Intermediate Working	Refrig. Refrig. Refrig. Refrig. Refrig. Refrig.	Semiannual Monthly Biweekly Semiannual Monthly Biweekly
GC/MS (non-VOA)	Various	Mix Solution	Frez/Ref	Working	Frez/Ref	Semiannual
GC/MS (VOA)	Various (Supelco)	Mix Solution	Freezer	Working	Freezer	Biweekly

Note: RT = Room Temperature
IR = Infrared
Frez = Freezer
Ref = Refrigerator

Source: ESE.

Table 9-2. List of Laboratory Instruments

Analysis Type	Number	Instrument
Gas Chromatography/ Mass Spectrometry:		
Semivolatiles	1	HP 5971 GC/MS with a HP 5890 GC capillary direct with HP-7673 Autosampler[1]; instrument uses a HP/UX computer system for data acquisition and reduction.
	2	EXTREL ELQ-400 MS with Varian 3400 capillary direct GCs with Leap A-200S Autosamplers; both instruments have a HP/UX computer system for data acquisition and reduction.
Volatiles	4	EXTREL ELQ-400 MS with Varian 3400 capillary direct GCs with jet separator interface; each instrument is attached to a Tekmar 2000 liquid sampler (LCS) and Tekmar 2016 and 2032 sixteen position autosamplers (ALS); all four instruments have a HP/UX computer system for data acquisition and reduction.
Gas Chromatography	3	HP 5890 GC configured for automatic sampling and equipped with dual Electron Capture Detectors. The GCs are attached to a personal computer (PC) via a Perkin Elmer interface with the Perkin Elmer (PE) Turbochrome 4.0 chromatography data system.
	1	Varian 3400 GC configured for automatic sampling and equipped with dual Electron Capture Detectors. The GC is attached to a personal computer (PC) via a Perkin Elmer interface with the Perkin Elmer (PE) Turbochrome 4.0 chromatography data system.
	1	HP 5890 GC configured for automatic sampling and equipped with dual Nitrogen-Phosphorus Detector. The GC is attached to a personal computer (PC) via a Perkin Elmer interface with the Perkin Elmer (PE) Turbochrome 4.0 chromatography data system.
	2	HP 5890 GCs configured for automatic sampling and equipped with Photoionization and ELCD Detectors. Attached are OI Purge & Trap Autosamplers capable of 16 positions. The GCs are attached to a personal computer (PC) via a Perkin Elmer interface with the Perkin Elmer (PE) Turbochrome 4.0 chromatography data system.
	1	HP 5890 GC configured for automatic sampling and equipped with Photoionization and Flame Detectors. Attached is an OI Purge & Trap Autosampler capable of 16 positions. The GC is attached to a personal computer (PC) via a Perkin Elmer interface with the Perkin Elmer (PE) Turbochrome 4.0 chromatography data system.
	1	Varian 3400 GC configured for automatic sampling and equipped with Photoionization and Flame Detectors. Attached is an OI Purge & Trap Autosampler capable of 16 positions. The GC is attached to a personal computer (PC) via a Perkin Elmer interface with the Perkin Elmer (PE) Turbochrome 4.0 chromatography data system.

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

Analysis Type	Number	Instrument
	1	Varian 3400 GC configured for automatic sampling and equipped with a Photoionization Detector. Attached is a Tekmar LSC2000 Purge & Trap Autosampler capable of 16 positions. The GC is attached to a personal computer (PC) via a Perkin Elmer interface with the Perkin Elmer (PE) Turbochrome 4.0 chromatography data system.
	2	Varian 3400 GC configured for automatic sampling and equipped with a Flame Ionization Detector. One GC is attached to a Waters ExpertEASE Chromatography Data Acquisition System operating under a VAX 3300 computer. The other is attached to a personal computer (PC) via a Perkin Elmer interface with the Perkin Elmer (PE) Turbochrome 4.0 chromatography data system.
	1	Varian 3410 GC configured for automatic sampling and equipped with FID Detectors. The GC is attached to a personal computer (PC) via a Perkin Elmer interface with the Perkin Elmer (PE) Turbochrome 4.0 chromatography data system.
	1	Varian 3400 GC configured for automatic sampling. One GC is equipped with a dual Thermal Selective Detector and FID. The GC is attached to a personal computer (PC) via a Perkin Elmer interface with the Perkin Elmer (PE) Turbochrome 4.0 chromatography data system.
	1	PE Sigma 2b GC with a Flame Ionization Detector. The GC is attached to a Waters ExpertEASE Chromatography Data Acquisition System operating under a VAX 3300 computer.
	1	PE Sigma 3b GCs. The GC is equipped with a Flame Ionization Detector and a Tekmar ALS 4200 Autosampler. The GCs are attached to a Waters ExpertEASE Chromatography Data Acquisition System operating under a VAX 3300 computer.
HPLC	1	Waters 600E powerline Gradient HPLC Systems equipped with a Fluorescence Detector and is attached to a personal computer (PC) via a Perkin Elmer interface with the Perkin Elmer (PE) Turbochrome 4.0 chromatography data system. The system is capable of post column derivitization.
	1	Waters 600E powerline Gradient HPLC System equipped with Fluorescence & Diode Array and 484 UV Detectors and post column derivitization; system is attached to a personal computer (PC) via a Perkin Elmer interface with the Perkin Elmer (PE) Turbochrome 4.0 chromatography data system.
	2	Waters 600E powerline Gradient HPLC System equipped with a scanning Fluorescence and UV Detector and attached to a personal computer (PC) via a Perkin Elmer interface with the Perkin Elmer (PE) Turbochrome 4.0 chromatography data system.
GPC	1	Waters Milli-Lab Gel Permeation Chromatograph (GPC).
	1	Zymark Benchmate with Waters Gel Permeation Chromatograph (GPC).
Metals	1	Jarrel-Ash 61E Inductively Coupled Plasma (ICP) Emission Simultaneous Spectrophotometer System with automated sampling accessories.
	1	Perkin-Elmer 5500B Inductively Coupled Plasma (ICP) Emission Sequential Spectrophotometer System.

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

Analysis Type	Number	Instrument
	1	Perkin-Elmer Model 4100ZL Atomic Absorption Spectrophotometer System equipped with Graphite Furnace, Zeeman Background Correction and automated sampling accessories.
	1	Perkin-Elmer 5100 Atomic Absorption Spectrophotometer System equipped with Graphite Furnace, Zeeman Background Correction and automated sampling accessories.
	1	Perkin-Elmer 3030 Atomic Absorption Spectrophotometer System equipped with Graphite Furnace, Zeeman Background Correction and automated sampling accessories.
	1	Leeman Model PS200 Cold Vapor Mercury Analyzer with automated sampling accessories.
	1	Varian Spectra AA-30 Atomic Absorption Spectrophotometer System equipped with a Cold Vapor Mercury Analyzer and automated sampling accessories. (Dedicated to Mercury analysis.)
	1	Perkin-Elmer Model 5000 Atomic Absorption Spectrophotometer equipped with a Flame accessory, D_2 background correction.
	2	CEM MSD 2100 Microwave Digestion Systems with automated accessories.
Inorganics	1	Astro Model 2001 System 2 TOC Analyzer.
	1	MCI Model TOX-10 TOX Analyzer.
	1	Dionex 2000I/SP Ion Chromatograph.
	1	Lachat Model 1200 Autoanalyzer with autosampler.
	2	Orion EA940 and 720A Specific Ion Meters.
	2	Milton Roy Model 301Visible Spectrophotometers.
	1	Buck Model HC404 IR Spectrophotometer.
	1	Hach Model DR13 Visible Spectrophotometer.
	1	Parr Model 1241 Adiabatic Bomb Calorimeter.
	1	HF Scientific Turbidimeter.
	1	Hach 2100A Turbidimeter.
	2	Hach COD Reactors.
	4	YSI Model 58 DO Meters.
	1	Tecator Soxtec Extraction Unit.
	1	Jenco Electronics, LTD. Model 1671 Conductivity Meter.
	3	Mettler PJ300 (1), Mettler AE240 (1), and Mettler AE163 (1) Analytical Balances.
	8	Ohaus GA200D(1), Ohaus AP1105 Plus(1), Ohaus E4000D(3), Ohaus E400(1), Ohaus C305S(2) Analytical Balances.
	2	American Scientific Products Z-3000-DR Top Loader (1) and SP180 Analytical Balance (1).

Specific calibration requirements for major classes of analytical procedures are described in Section 9. If the calibration requirements of the specified analytical method are more stringent than the procedures described in this CQAP, the method procedures will be followed.

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

Single Point Calibration--Single point calibration is a viable alternative to a calibration curve for gas chromatography drinking water methods. (This is not applicable to gas chromatography volatile drinking water methods.) Single point standards are prepared from the secondary standard dilutions. The single point standard is prepared at concentration that produces a response that deviates no more than twenty percent from the sample extract response. The single point calibration is only used when analytes of interest are below the specified reporting limits. If an analyte of interest is detected above the reporting limit, the sample is reanalyzed using a three point standard curve calibration. The procedures for a standard curve calibration are presented below.

Standard Curve Calibration--Initial calibration standard solutions are prepared by serial dilutions of a single stock standard solution to cover the analytical working range of the method. These are either composite standards of more than one analyte or single-analyte solutions. The concentrations are 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 desired working range are prepared and analyzed with a blank. A medium level standard and a blank are analyzed at the beginning of each continuing analytical run. At least one calibration standard at the middle or high range of the curve is analyzed every 10 samples and repeated at the end of the run.

The initial calibration curve is 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 are expressed in terms of the concentration of the standard solution, because 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 standard response factors are within 15 percent of the inital calibration for the EPA SW-846 gas chromatography methods, 10 percent for the EPA 600 series gas chromatography methods, 20 percent for drinking water gas chromatography methods, and 10 percent for HPLC methods. Data is not rejected due to an ending standard that fails QC requirements, and
4. 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 is 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.

9.3.2 GAS CHROMATOGRAPH (GC-VOLATILES) CALIBRATION

Standard Curve Calibration--Calibration standard solutions are prepared as needed by dilutions of several intermediate standard solutions, covering the analytical working range of the method. These are either composite standards of more than one analyte or single-analyte solutions. The concentrations are 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 are prepared and analyzed with a blank. At least one calibration standard at the middle to high range of the curve is analyzed every 10 samples. GC-volatile methods do not require an ending standard. Calibration is the same 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 is practiced to ensure the instrument is calibrated and in proper working condition. The GC/MS is 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.

GC/MS Calibration--Relative response factors for the individual compounds is 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.

An initial calibration with a minimum of three points (or the number of standards per method requirements) is analyzed before samples are analyzed to determine the instrument linearity. The average response factor (RF) is calculated for each compound. The response factors for the System Performance Check Compounds (SPCCs) are $\geq$ 0.300 except for bromoform which is $\geq$ 0.250 for EPA 624, EPA 8240, and EPA 8260. The percent relative standard deviation (%RSD) is calculated from the response

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

Key Ions	Ion Abundance Criterion
For DFTPP*	
51	30 to 60 percent of mass 198
68	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
For BFB*	
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.

factors of each calibration check compound (CCC). Reponse factors are within 30 percent relative standard deviation for EPA 624, EPA 8240, and EPA 8260. The percent relative standard deviation for the remainder of the compound list is a maxmum of 40 percent. For EPA 524.2, the initial calibration is within 20 percent relative standard deviation for all compounds. For EPA 8270, the initial calibration is within 30 percent relative standard deviation for the CCCs. The response factors for the SPCCs are $\geq$ 0.050. For EPA 625, the initial calibration is <35 percent relative standard deviation for all compounds.

A 1-point calibration using a midlevel standard from the initial calibration is used daily for all subsequent analysis, except for Method 524.2 where the analytes are quantitated directly from the calibration curve. For EPA 624, EPA 8240, and EPA 8260, the CCCs are within 25 percent difference of the average response factor of the initial calibration. The SPCCs have the same criteria as the initial calibration. For EPA 524.2, the CCCs are within 30 percent difference of the average response factor of the initial calibration. For EPA 8270, the CCCs and SPCCs have criteria as the initial calibration. For EPA 625, the CCCs are within 20 percent difference of the average response factor of the initial calibration. 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, (in addition, fluorobenzene for 524.2) for volatiles (EPA 624 and 8240); and 1,4-dichlorobenzene-d4, naphthalene-d8, acenaphthene-d10, phenanthrene-d10, chrysene-d12, and perylene-d12 for semivolatiles (EPA 625 and 8270). A retention time and response check is performed on every internal standard for samples that are analyzed.

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, ultraviolet (UV) absorption, turbidimetric] that use a standard curve for calibration [except total organic carbon (TOC), chemical oxygen demand (COD), infrared (IR), and potentiometric]. Working standard solutions are prepared by serial dilution of a single-stock standard to bracket the analytical working range of the method. Working standard solutions are either composite standards of more than one analyte or single-analyte solutions. The standard concentrations are 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 are prepared and analyzed with a blank. A continuing working standard and a blank are analyzed, at a minimum, at the beginning of every analytical run; and at least one midlevel standard, which is the continuing calibration verification (CCV) standard, is 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, the standards are from the same supplier, but from a different lot. Certificates of Analysis are available for all standards.

The working curve is 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 during the run and at the end of the run is within 20 percent of true value
4. The ICV is within 20 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 is 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 are prepared to include the analytical working range of the method; these solutions are either composite standards of more than one metal or single-metal solutions. The standard concentrations are 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 are prepared and analyzed with a blank. The calibration standards and the blank are analyzed at the beginning of every analytical run, and at least one midlevel standard is analyzed at minimum intervals of every 20 samples 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.

The working curve is 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 during the run and at the end of the run, is within 20 percent of true value;
4. The ICV is within 10 percent 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 a trace metal in the sample is obtained by entering the response for the sample into the working calibration curve equation and determining the metal concentration in the digestate. The value is corrected by the appropriate digestate volume, sample size, applicable dilution factor, and moisture content (for soils) to generate a final sample concentration.

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. 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 during the run and at the end of the run to document constant instrument response. This solution is in the midrange 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; and
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)]:

Weight (g)	Tolerance Limits
0.1	± 0.0005
0.5	± 0.0005
1.0	± 0.0005
3.0	± 0.0005

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

Weight (g)	Tolerance Limits
5	± 0.02
20	± 0.05
50	± 0.05

The calibration results are recorded in the appropriate balance 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. Two sets of Class S weights are available in-house. Qualified service personnel calibrate the analytical balances semiannually, utilizing Class S and NIST certified weights. The semiannual calibration is documented by a tag on the instrument. Service calibration records are kept on file in the laboratory.

9.3.7 TITRIMETRIC METHODS CALIBRATION

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

9.3.8 TOC CALIBRATION

The TOC analyzer is calibrated with a prepared standard using a single-point calibration. The standard is analyzed before beginning every analytical run. 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 ±15 percent of true value.

9.3.9 COD CALIBRATION

Prepared standards are used to verify the 0- and 500-mg/L readings with the standard curve. The standard curve is developed by Hach Chemical Company for COD on a spectrophotometer using 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 daily when in use.

9.3.11 TOTAL ORGANIC HALIDES (TOX) CALIBRATION

The TOX analyzer is calibrated with a prepared standard 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 ± 15 percent of true value.

9.3.12 pH CALIBRATION

The pH meter is calibrated with three buffer solutions at pH 4, pH 7, and pH 10 prior to use. The pH meter temperature selector is set to ambient temperature. The probe is placed on the pH 7 buffer; and the calibration switch is adjusted until it reads 7.00 units. The initial pH value and the buffer adjusted value should be recorded. The procedure is repeated with the pH 4 and pH 10 buffer solutions. The pH of the pH10 buffer should be 10 ± 0.05 units; if not, the pH probe and internal solution is checked and the calibration procedure is repeated.

9.3.13 SPECIFIC CONDUCTIVITY CALIBRATION

The instrument is calibrated with 0.01 M and 0.10 M KCL solutions. The conductivity reading of the 0.01 M KCL must be 1,413 umhos ± 15%; the 0.10 M KCL must be 12,900 units ± 15%. If the calibration standards are outside the acceptance criteria, new standards are prepared and the instrument is recalibrated.

9.3.14 PENSKY-MARTENS CLOSE-CUP TESTER CALIBRATION

The ignitibility of the p-xylene standard is determined prior to use of the Pensky-Martens Close-Cup Tester. The standard should ignite at 27.2 ± 1°C. If not, the condition and operation of the apparatus is checked, especially the tightness of the lid, the action of the shutter, and the position of the test flame. After adjustment, the test is repeated with the p-xylene standard. The barometric pressure is read and recorded at the time of analysis.

9.3.15 DISSOLVED OXYGEN CALIBRATION

The dissolved oxygen probe is 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. The temperature is read and recorded at the time of analysis.

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 9-4 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.

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 Department Manager is responsible for overseeing the performance of the analysis in accordance with the defined quality control practices outlined in this CQAP.

For all analyses performed by ESE, the QC checks described in this section are *mandatory unless alternate procedures* are given in a specific project QA Plan or otherwise agreed upon by the Laboratory Manager and the Project Manager. 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 are followed. Sections 5.0 and 9.0 contain QC evaluation criteria for laboratory methods and calibrations. Section 11.2 describes precision and accuracy calculations used for control samples. Laboratory Department Managers are responsible for reviewing QC criteria for each method performed by their department. Permanent changes to the acceptance criteria are approved by the Department Managers, Operation Managers, and QA/QC Coordinator and are incorporated into this document in accordance with Section 3.3. Project-specific revisions are documented in a specific project QA Plan.

For QC purposes, a Sample Delivery Group (SDG) is used to identify a group of samples to be received by the laboratory from a client. The SDG is a set of twenty or fewer environmental samples by matrix (e.g. soil, water, etc.) received by the laboratory from a client over a period of up to fourteen calendar days or seven calendar days if a fourteen day turnaround time is requested. (Data from all samples in a SDG are due on the same date.) If a SDG is not indicated by the client, the number of samples extracted and/or

Table 11-1. Minimum QC Sample Requirements

QC Sample	CLASS™ Code	Frequency	Analysis
Method Blank	MB	Daily or 1 per 20 samples or SDG	All analyses
Standard Spike */ Laboratory Control Sample	SP	Daily or 1 per 20 samples or SDG	All analyses except (a)
Sample Matrix Spike**	SPM1	Daily or 1 per 20 samples or SDG (b)	All analyses except (a)
Sample Matrix Spike Duplicate	SPM2	Daily or 1 per 20 samples or SDG	All analyses except (a)
Surrogate***	SUR	All samples (organics only)	Required for all organic samples and standards, when required
Replicate	RP	Daily or SDG	For miscellaneous inorganic parameters (a)
Analytical Spike	SPX	10% of samples or as specified by the method	Required for GFAA and CVAA methods only
Serial Dilution	SD	If SPM fails acceptance criteria only	Required for ICAP only

(a) Miscellaneous inorganic parameters including: conductivity, pH, residues, DO, % moisture, turbidity, etc.

(b) TCLP, 5% or 1 per waste type, whichever is greater. Sample Matrix Spike Duplicate not required for this analysis.

SDG Sample Delivery Group

* Standard 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 and soil samples. 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.

** Sample Matrix Spike is a spike into a sample matrix which is carried through sample preparation, sample digestion, or extraction to sample analysis.

*** Surrogates are required for all organic methods as appropriate.

prepared for instrumental analysis as one group in one 24-hour period constitute an extraction group. The number and type of QC samples specified in Section 11.0 apply to either a SDG or an extraction group, if a SDG is not specified. For example, a group of samples that is extracted on the same day and (if required) undergoes concentration and cleanup procedures on subsequent days are considered one extraction sample group 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 determines the number of samples per sample group for QC purposes. The number and type of QC samples specified in Section 11.0 also apply to this group of samples.

When required, as for a specific project, the Department Manager may insert into a current sample batch either spiked sample or sample duplicate results of a previously analyzed sample for QC purposes (with all previous batch references documented in the current batch folder). The Department Manager reviews the results of the previous sample batch to ensure that the analysis meets QC criteria for the current project.

Blind QC check samples are samples of known composition by the QA/QC Coordinator, USEPA, etc., but of unknown composition to the analyst. Blind QC check samples from the USEPA are analyzed by the laboratory semiannually to evaluate the laboratory's analytical performance. If the blind QC check sample data are not acceptable, a corrective action summary report is written and submitted to appropriate states and agencies for certification requirements.

A sample matrix spike (SPM1) is defined as an environmental sample to which known concentrations of control analytes have been added. In addition, if enough sample is present, the sample is split into a duplicate, known as a matrix spike duplicate (SPM2). Sample matrix spikes are included in batch QC for all analyses except miscellaneous inorganic parameters such as pH, residues, dissolved oxygen, % moisture/solids, conductivity, and turbidity. Results of the sample, and SPM1/2 pair are used to generate recoveries. This data is used to assess the accuracy of the analytical procedure (percent recovery) and indicate and matrix interferences. SPM1/2 results are also used to asses the precision (relative percent difference) of the analytical procedure. Selection of the sample to be spit and spiked is specified by the client or the laboratory. Results are reported on a per batch basis.

Control spikes (standard matrix spikes (SP) or QC check standards) are placed into blank matrices for all analyses except miscellaneous inorganic parameters such as pH, residues, dissolved oxygen, % moisture/solids, conductivity, and turbidity. This spike is used for method control and verifies the calibration standards, if an ICV is not analyzed. A sample replicate is prepared and analyzed for inorganic parameters such as pH, residues, dissolved oxygen, % moisture/solids, conductivity, and turbidity. The relative percent difference between the sample and the replicate is used to assess analytical precision.

It is ESE's policy to control sample analyses with QC criteria that are under the control of the technicians and analysts utilizing the analytical procedure. Therefore, emphasis is placed on calibration, method blanks, and standard matrix spike results. When these QC sample results are within criteria, acceptable method performance is documented. Sample matrix spikes are reported and evaluated for precision and accuracy, but not necessarily used for method control. A sample matrix spike that has recoveries outside of QC criteria is evaluated against other available QC data, within the batch, to determine of the method is in control and if sample flagging is warranted. The failure of a sample matrix spike to achieve acceptable QC criteria when a standard matrix spike in the same batch has acceptable recoveries, indicates whether or not the sample matrix interferes with the quantitation of the target analytes. Cases where poor precision or erratic recoveries are seen indicate that the analysis method selected for the samples may not be appropriate for that matrix type, not that the method is out of control.

Precision and spike recovery checks are discussed in further detail in Section 11.2.

11.1.1 GC/MS MINIMUM QC

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

1. All samples spiked with surrogate.
2. At least 5 percent spikes in a sample matrix (SPM1) with selected analytes and surrogates are analyzed.
3. At least 5 percent duplicate spikes in a sample matrix (SPM2) with selected analytes and surrogates are analyzed.
4. At least 5 percent QC standard spikes (SP) in a blank matrix with selected analytes and surrogates are analyzed.
5. At least 5 percent method blanks spiked with surrogates are analyzed.
6. An initial calibration with a minimum of three points (or the number of standards per method requirements) is analyzed before samples are analyzed. Response factors for the Calibration Check Compounds (CCCs) are within 30 percent relative standard deviation for EPA 624, EPA 8240, and EPA 8260. The response factors for the System Performance Check Compounds (SPCCs) are ≥ 0.300 except for bromoform which is ≥ 0.250. The percent relative standard deviation for the remainder of the compound lists is a maximum of 40 percent. For EPA 524.2, the initial calibration is within 20 percent relative standard deviation for all compounds. For EPA 8270, the initial calibration is within 30 percent relative standard deviation for the CCCs. The response factors for the SPCCs are ≥ 0.050. For EPA 625, the initial calibration is < 35 percent relative standard deviation for all compounds.
7. Instrument tuning protocols are performed and are within criteria (listed in Section 9) prior to analysis.
8. Continuing calibration standard is analyzed at a frequency of 5 percent or at the beginning of a daily continuing analytical sequence. For EPA 624, EPA 8240, and EPA 8260, the CCCs are within 25 percent difference of the average response factor of the initial calibration. The SPCCs have the same criteria as the initial calibration. For EPA 524.2, the CCCs are within 30 percent difference of the average response factor of the initial calibration. For

EPA 8270, the CCCs and SPCCs have the same criteria as the initial calibration. For EPA 625, the CCCs are within 20 percent difference of the average response factor of the initial calibration.

9. Detection limits for each parameter are determined and checked to ensure they meet reporting limit requirements specified for the project.
10. Samples are within the concentration range of the standards.

11.1.2 GC AND HPLC MINIMUM QC

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

1. All samples spiked with surrogate(s), if specified by the method.
2. At least 5 percent spikes in a sample matrix (SPM1) with selected analytes and surrogate(s) (if applicable) are analyzed.
3. At least 5 percent duplicate spikes in a sample matrix (SPM2) with selected analytes and surrogate(s) (if applicable) are analyzed.
4. At least 5 percent QC standard spikes (SP) in a blank matrix with selected analytes and surrogate(s) (if applicable) are analyzed.
5. At least 5 percent method blanks spiked with surrogate(s) (if applicable) are analyzed.
6. A minimum of three standards or the number of standards specified by the method are analyzed as a standard curve except for non-volatile drinking water methods where single point calibration, as described in Section 9, is applicable.
7. Correlation coefficient of the standard curve is equal to or greater than 0.995.
8. Samples are within the concentration range of the standards.
9. Midlevel calibration standards are repeated at minimum intervals of every 10 samples and at the end of a run (except GC-volatiles), and response factors are within 15 percent of the initial calibration for the EPA SW-846 gas chromatography methods, 10 percent for the EPA 600 series gas chromatography methods, 20 percent for drinking water gas chromatography

methods, and 10 percent for HPLC methods. Data is not rejected due to an ending standard that fails QC requirements. GC-volatile methods do not require an ending standard; midlevel calibration standards are analyzed.

10. Detection limits for each parameter are determined and checked to ensure they meet reporting limit requirements specified for the project.

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 apply, except for CLP SOW:

1. At least 5 percent spikes in a sample matrix (SPM1) with selected elements are analyzed.
2. At least 5 percent duplicate spikes in a sample matrix (SPM2) with selected elements are analyzed.
3. At least 5 percent QC standard spikes (SP) in a blank matrix with selected elements are analyzed.
4. At least 5 percent method blanks are analyzed.
5. A minimum of three standards or the number of standards specified by the method are analyzed as a standard curve.
6. Correlation coefficient of the standard curve is equal to or greater than 0.995.
7. Samples are within the concentration range of the standards (or of the ICAP instrument).
8. Midlevel calibration standards are repeated at minimum intervals of every 20 samples and at the end of a run. Response of the elements are within 20 percent of the true value for CVAA and GFAA (10 percent for ICAP).
9. Detection limits for each element are determined and checked to ensure they meet reporting limit requirements specified for the project.

11.1.4 MISCELLANEOUS METHODS MINIMUM QC

For each batch of samples analyzed by ion chromatographic, colorimetric, spectrophotometric, IR, UV absorption, and titrimetric methods (except for additional miscellaneous inorganic methods such as pH, residues, dissolved oxygen, % moisture/solids, conductivity, turbidity), the following QC checks apply:

1. At least 5 percent QC standard spikes (SP) in a blank matrix are analyzed.
2. At least 5 percent spikes in a sample matrix (SPM1) are analyzed.
3. At least 5 percent duplicate control spikes in a sample matrix (SPM2) are analyzed.
4. At least 5 percent method blanks are analyzed.
5. A minimum of three standards or the number of standards specified by the method are analyzed as a standard curve.
6. Correlation coefficient of the standard curve is equal to or greater than 0.995.
7. Samples are within the concentration range of the standards.
8. Midlevel calibration standards are repeated at minimum intervals of every 20 samples and at the end of a run. Responses of the standards are within 20 percent of the true value.
9. Detection limits for each parameter are determined and checked to ensure they meet reporting limit requirements specified for the project.

For each batch of samples analyzed for additional inorganic parameters such as pH, residues, dissolved oxygen, % moisture/solids, conductivity, turbidity, the following QC checks apply:

1. At least 5 percent sample replicates are analyzed.
2. At least 5 percent method blanks are analyzed.
3. Continuing calibration standards, if applicable, are analyzed at a frequency of 5 percent.
4. Detection limits for each parameter are determined and checked to ensure they meet reporting limit requirements specified for the project.

11.2 ROUTINE METHODS USED TO ASSESS PRECISION AND ACCURACY

11.2.1 PRECISION

Precision is the degree of mutual agreement among individual measurements repeatedly performed utilizing the same test procedure and conditions. Precision is assessed for applicable parameters by calculating the RPD of two duplicate spike samples as follows:

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

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 Section 5 of the CQAP. Additionally, the spike levels used to determine the precision targets are listed in Section 5.

11.2.2 ACCURACY

Accuracy is the degree of agreement between a sample's target value (true or expected 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:

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

The following equation is an example calculation:

1 mL of spike with concentration of 100 ppb

10 mL of sample with concentration of 10 ppb

spiked sample concentration of 20 ppb

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

Each calculated R value is compared to accuracy criteria listed in Section 5. The accuracy ranges provided in Section 5 are based on the mean accuracy measured or expected, as from method criteria, for each parameter plus or minus three standard deviations of the mean. The spike levels used to determine the accuracy targets are listed in Section 5. If RPD or R values for standard spikes within a batch do not meet acceptance criteria specified in Section 5, the batched samples are re-analyzed or sample results are flagged appropriately. If nonconformances occur, the Department Manager or designee is notified and necessary corrective action taken. Proper corrective action procedures are described in Section 13.

11.2.3 EVALUATION OF CONTROL CHARTS

Control charts are graphical plots of analysis results that illustrate statistical control by monitoring trends in a measurement process through time or sequence of analysis. By monitoring the measurement process, control limits are generated to demonstrate that the method is statistically in control. It is improbable that a point could lie outside the limits on a control chart while the system remains in a state of control.

Analysts have the ability, through the ESE Laboratory Information Management System CLASS™ to generate control limits using historical ESE data. If sufficient in-house data is unavailable, control limits are derived from published USEPA method data, if available. Control limits are updated yearly or as needed using historical ESE data.

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

$$UCL\text{-}_x = \bar{X} + 3SD$$

$$UWL\text{-}_x = \bar{X} + 2SD$$

$$LWL\text{-}_x = \bar{X} - 2SD$$

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

where: 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 control limits are specifically tabulated according to matrix and QC type.

An analysis is considered out of control when any one of the following situations exist:

1. One point plots outside the control limits,
2. Eight consecutive points plot on the same side of the mean,
3. A systematic pattern is evident,
4. Three consecutive points plot within the control limits but outside the warning limits.

The occurrence of any of these events is investigated and corrective actions are taken as required to return the system to a state of statistical control. Corrective actions are documented using the appropriate corrective action form, Section 13.

11.3 METHOD DETECTION LIMITS AND PRACTICAL QUANTITATION LIMITS

11.3.1 METHOD DETECTION LIMITS (MDLs)

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

Minimally, MDL studies are performed annually for methods routinely used by the laboratory.

The detection limit is defined (40 CFR, Part 136 Appendix B) 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

$t_{(n-1,\ 1-\alpha,\ =\ 0.99)}$ = Students t-value appropriate to a 99-percent confidence level and a standard deviation estimate with n-1 degrees of freedom.

Instrument Detection Limits (IDL) are calculated similarly to the MDLs. Instead of the detection limit study being performed in a sample matrix that has gone through the appropriate extraction or digestion procedure, IDLs are generated by repetitively analyzing standard matrix spikes in the same manner discussed in the 40 CFR Part 136 Appendix B.

11.3.2 PRACTICAL QUANTITATION LIMIT (PQL)

The PQL is the lowest concentration of an analyte that can be reliably achieved within a specified degree of precision and accuracy throughout routine laboratory conditions. The PQL is defined as approximately three to five times the Method Detection Limit. The PQL or reporting limit may be modified to meet clients' specifications.

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 uses the company developed Laboratory Information Management System, CLASS™, for all projects.

If applicable, example calculations are included with the summarized data to facilitate review. The entry of input data and calculations are checked and the signature/initials of the analyst or individual entering the data and reviewer(s) accompany all data transferred (with and without data reduction). All final analysis results are calculated according to the referenced methods specified in Section 5.

For routine analyses performed at the Peoria Laboratory, sample response data is 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, and
4. Sample concentrations.

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

Linear: Concentration = Intercept + M (Response)

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

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

Purgeable organics by GC/MS are calculated as follows:

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

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),

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:

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

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),

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

volume = volume of extracted sample (mL), and

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

The final data for GC/MS semivolatiles and volatiles analyses are calculated by the computer data acquisition system attached to each mass spectrometer.

QC acceptance criteria (Section 5) for the relative percent difference of replicate spike recoveries and the range of acceptable spike recoveries are electronically stored in the computer data management files for each STORET number/method code combination. If the samples in a batch (sample group) do not pass all the QC checks (Section 11), the results reported for all samples processed in the same sample group are considered as suspect and flagged if appropriate; analyses may need to be repeated.

Completed batch folders are stored in a secured central location arranged by departments and numerically by batch number. Chromatograms, copies of parameter notebooks, and all other pertinent raw data and other documentation are stored in the batch folders.

Once the data set is complete for each sampling effort, the Project Manager organizes the information for final report format, according to project requirements. The Project Manager is responsible for final QC review and release of the data.

12.1.1 THE DOCUMENTATION RECORDS

All manual documentation of raw data is done in notebooks or appropriate forms. All notebooks used have consecutively numbered pages. All notebook entries are made in indelible ink. Any blank portions of data forms or notebook pages are lined out with black ink and initialed by the analyst.

12.1.1.1 GC/HPLC

Extraction Logbook--An extraction logbook copy, filled out by the analyst performing the sample extraction, accompanies each lot of samples throughout analysis. This sheet includes at least the following data:

1. Project name,
2. Extractor's initials,

3. Type of sample matrix,
4. Field group name,
5. Sample numbers,
6. Date extracted,
7. Analytical method,
8. Initial volume or wet weight of sample extracted,
9. Initial pH (water sample),
10. Extracting solvent,
11. Final volume/solvent,
12. Extract box identification,
13. Date of cleanup (if required),
14. Notes and comments affecting the extraction procedure, and
15. Surrogate/spike preparation reference number and spike volume.

After extraction is complete, the extraction logbook copies accompany the sample arrival notice to the instrumental analyst. The extracted samples are refrigerated and stored in boxes, in a central location, until the required analysis. The box number is referenced on the extraction logbook copy. Each extract vial is properly labeled and include the following information:

1. Project name,
2. Field group name,
3. Sample number,
4. Analyte group and matrix,
5. Date extracted, and
6. Extraction logbook reference number.

Instrument Logbooks--During analysis, the following information is recorded in an instrument notebook:

1. A log of the types of analyses run on the instrument, to include:

a. Column/instrument conditions and temperature zones,

b. Sample numbers or other identification of samples,

c. Reference to a method or analyte group describing the analysis,

d. Sequence date and analyst initials,

e. Detector used [e.g., flame ionization detector (FID)] (on cover), and

f. Detector conditions.

2. Service records are kept in a separate maintenance log.

Chromatograms--The analyst will include the following information on the chromatogram (if not automatically printed):

1. Date and time of analysis,
2. Analyst identification,
3. Instrument used,
4. Field group name,
5. Sample number and other identification for each chromatogram, and
6. Concentration/dilution factor for each sample (not for GPC).

After the analysis and data reduction are complete, the chromatograms are stored in the batch file folder and the data entered into CLASS™. The folder is submitted to Laboratory Information Services for storage in the secured central filing location.

Standards--Prior to analysis, stock standard solutions and working solutions covering the working range of the method are prepared. Procedures used in preparing the standards are recorded in standard preparation logbooks. The following information is recorded:

1. Reference standard source,
2. Lot number,
3. Date of preparation,
4. Analyst's name or initials,
5. Actual weight measured,

6. Volumetric flask volume,
7. Calculated concentration,
8. Solvent name and lot number,
9. Dilutions, and
10. Expiration date.

Immediately after an analytical standard has been prepared, the standard is transferred to an amber glass vial, bottle, or appropriate container and properly labeled. Standards are refrigerated when not in immediate use.

12.1.1.2 GC/MS

Extraction Logbook--Once a batch has been established, the sample extraction and analysis procedure begins. An extraction logbook copy, filled out by the analyst performing the sample extraction, accompanies each lot of samples throughout analysis. This sheet includes at least the following data:

1. Project name,
2. Extractor's initials,
3. Type of sample matrix,
4. Field group name,
5. Sample numbers,
6. Date extracted,
7. Analytical method,
8. Initial volume or wet weight of sample extracted,
9. Initial pH (water sample),
10. Extracting solvent,
11. Final volume/solvent,
12. Extract box identification,
13. Date of cleanup (if required),
14. Notes and comments affecting the extraction procedure, and

15. Surrogate/spike preparation reference number and spike volume.

After extraction is complete, the extraction logbook copies accompany the sample arrival notice to the instrumental analyst. The extracted samples are refrigerated and stored in boxes, in a central location, until the required analysis. The box number is referenced on the extraction logbook copy. Each extract vial is properly labeled and include the following information:

1. Project name,
2. Field group name,
3. Sample number,
4. Analyte group and matrix,
5. Date extracted, and
6. Extraction logbook reference number.

Spectral Data and GC/MS Computer Quantitation Report--The quantitative sample and standard data generated by the GC/MS data system and all mass spectral information are labeled and placed in the batch file folder. Manual data reduction is indicated by the flag "M" on the quantitation report.

Standards--Prior to analysis, stock standard solutions and working solutions covering the working range of the instrument are prepared. Procedures used in preparing the standards are recorded in standard preparation logbooks. The following information is recorded:

1. Reference standard source,
2. Lot number,
3. Date of preparation,
4. Analyst's name or initials,
5. Actual weight measured,
6. Volumetric flask volume,

7. Calculated concentration,
8. Solvent name and lot number,
9. Dilutions, and
10. Expiration date.

The analytical standard is transferred immediately after preparation to a properly labeled amber glass vial, bottle, or appropriate container. Standards are refrigerated when not in immediate use.

GC/MS Instrument Logbooks--Whenever the GC/MS is used for sample analysis, the following information is recorded in an instrument logbook:

1. Instrument conditions of the gas chromatograph,
2. Instrument conditions of the mass spectrometer,
3. Analyst's initials,
4. Date of sequence,
5. Sample number or other identification,
6. Dilution factor,
7. File reference number (FRN), and
8. Method reference.

Compound Identification--Compound identification is made in terms of the full-scan mass spectrum obtained in the electron impact mode at 70 electronvolts (eV). Compound identification requires the presence of all significant major ions at the appropriate relative abundance as obtained with an authentic compound or reference spectrum from a reputable literature source. The selection of significant ions is strongly compound dependent, and because of this and other considerations, the identification of compounds entails considerable professional judgment and experience.

The most convincing evidence for compound identification is comparison of spectrum with that of an authentic compound obtained under identical operation conditions. When this is not possible due to compound availability, computer identification or library search is used and flagged as tentative identification.

Compound Quantification--The technique of extracted ion current profiles is employed for the preliminary qualitative searching and for quantification of individual compounds. Appropriate internal standards are employed to permit quantification in terms of the relative response to these internal standards. Concentration calculations and data reduction procedures are given in Section 12.3.

Spiking with Internal Standards--All samples are spiked with quantitation standards prior to the GC/MS analysis. Appropriate internal standards are selected for the remaining categories.

GC/MS Instrumental Detection Limits--The instrumental detection limit refers to the least quantity of material required to provide a total mass spectrum, of sufficient quantity, to permit compound identification. The mass spectrum contains all major ions with the appropriate relative abundance within 20 percent of either an authentic compound analyzed under identical conditions or an appropriate reference spectrum from the literature.

Data Management--Raw data such as mass spectral chromatorgrams, as well as calculated results, are stored on magnetic tape. Various reports present the calibration, tune, and on-column/final results. Magnetic tapes are uniquely identified, with data stored sequentially, to allow easy retrieval. Final GC/MS data results are transmitted to CLASS™ by project and sample number. The analyst processes the transmitted data and generates a batch report. The batch folder, containing the quantification report, batch report, copies of logbooks, and other pertinent raw data is turned into Laboratory Information Services for storage in the secured central filing location.

12.1.1.3 Trace Metals

Digestion or Sample Preparation Logbook--A copy of the digestion or sample preparation logbook, filled out by the analyst performing the sample digestion or sample preparation, accompanies each lot of samples throughout the analysis. This logbook copy will include the following data:

1. Method used (GFAA, CVAA, ICAP)
2. Analyst's initials,
3. Date sample digested,
4. Initial volume or weight,
5. Final volume,
6. Spiking solution used and standards preparation reference number,
7. Field Group,
8. Sample numbers, and
9. Notes or comments affecting the digestion procedure.

For ICAP, the ICAP computer produces a data file that is evaluated and transmitted to CLASS™. The analyst then generates a batch for review. The batch folder containing the batch report, the data file, copies of logbooks, and all other pertinent raw data are submitted to Laboratory Information Services for storage in the secured central filing location.

Laboratory Logbooks--Each instrument has its own laboratory logbook. After each analysis, the analyst records the following information in the logbook:

1. Problems encountered during the analysis,
2. Comments about the samples and/or analytical procedure,
3. Instrument used,

4. Method used (GFAA, CVAA, ICAP),
5. Date of analysis,
6. Analyst(s),
7. Element,
8. Instrument conditions,
9. Preparation logbook reference number,
10. Preparation batch reference number, and
11. Sample numbers.

Standards--Stock standard solutions are purchased from vendors. These stock solutions are certified by the vendor for purity and concentration.

Standard preparations are recorded in a logbook. The information recorded includes preparer's name, lot number, date of preparation, volumes used, calculated concentrations, and dilutions.

Volumetric dilutions are made from the stock solution to obtain working solutions. Serial dilutions are then made from the working solutions to obtain working standards to be used to generate standard curves. Working standard solutions are stored in volumetric flasks and properly labeled with the following information:

1. Preparer's name or initials,
2. Date of preparation,
3. Element(s),
4. Concentration, and
5. Expiration date (if not prepared daily).

12.1.1.4 Inorganics

Raw data for most inorganic analyses is documented through the use of parameter logbooks. The logbooks may vary slightly in format dependent upon the type of analysis, but, at a minimum contain the following:

1. Analysis date,
2. Parameter,
3. Standard curve range and responses (where applicable),
4. Analytical batch number,
5. Instrument conditions (where applicable),
6. Method reference,
7. Sample, standard, QC sample and blank identification and responses or concentration as applicable and,
8. Analyst's initials.

Raw data for specialized instrumental analyses are documented in the following sections.

Inorganic Analysis by Autoanalyzer

After the data has been recorded in the parameter logbook, the raw data is placed in a batch file folder with copies of the notebook pages and any additional related information. These data are entered manually uploaded to CLASS™ to generate a uniquely numbered batch. The batch is reviewed for correctness and is submitted for review and finalization. When review and finalization are complete, the reviewer signs and submits the batch to Laboratory Information Services for storage in the secured central filing location.

Laboratory Logbooks--Each analytical parameter has its own laboratory logbook. During analysis, the following information is recorded:

1. Date of analysis,
2. Parameter,

4. Analytical batch number,
5. Method reference,
6. Instrument conditions,
7. Calibration standard setting and response,
8. Standard curve range, responses, and date of curve preparation,
9. Sample, standard, QC sample, and blank identification and responses or concentrations, and
10. Analyst's initials.

Inorganic Analysis by Ion Chromatography

Chromatograms--All information on the chromatograms from each analytical run is electronically recorded from the input provided during run set up. This information includes the following:

1. Analyst's initials,
2. Analytes,
3. Analysis date and time,
4. Instrument identification,
5. Integration parameters,
6. Sample, standard, and QC sample identification with concentrations and responses, and
7. Dilution factors when appropriate.

These data are manually entered into CLASS™ and an unique batch number is assigned. The data are reviewed by the analyst for correctness and submitted for review and finalization. When review and finalization are complete, the reviewer signs and submits the batch to Laboratory Information Services for storage in the secured central filing location.

Laboratory Logbooks-The instrument has its own laboratory logbook. The following information is recorded in the logbook during the set up of the analytical run:

1. Analysis date,
2. Analyte,
4. Instrument identification and operating conditions,
5. Calibration standards and preparation dates,
6. Notes and comments as appropriate, and
7. Sample and QC sample identification numbers with dilution factors when applicable.

12.2 DATA VALIDATION

Unless otherwise specified by the client, the following procedures for review/validation of data are employed.

12.2.1 LABORATORY ACTIVITIES

Data review is performed at the bench by the analyst. The analyst reviews preliminary data entries, calculations, holding times, precision and accuracy, and calibration checks. The analyst provides an explanation and/or corrective action for any method control parameters which are outside criteria and signs the analytical batch when ready to release the data for further processing and review. This information os relayed immediately to the Department Manager, who notifies the appropriate Project Manager and Laboratory QA/QC Coordinator.

The analyst's supervisor or a designated reviewer also reviews the analytical documentation associated with the batch (such as sample preparation/digestion/extraction logbook copies, instrument logbook copies, etc.) and any explanations or corrective actions provided by the analyst. The Department Manager or designee signs and finalizes the batch after the final review.

The Project Manager checks analytical data batches that have explanations and corrective actions. The Project Manager also reviews all final data reports for inconsistencies and completeness prior to releasing the reports to the client; qualification or flagging of data and/or QC summaries are provided as appropriate.

The Laboratory QA/QC Coordinator performs quarterly audits to check that required QC procedures are being followed. This procedure entails random review of analytical batches to see that the QC designated for the analysis is being consistently performed. A record of this audit is maintained by the QA/QC staff. The Laboratory QA/QC Coordinator has the capability to initiate and follow up on corrective actions to resolve QC problems.

The minimum QA/QC data that should be included in the data batch are the following:

1. Sample data (matrix, date of extraction, and date of analysis),
2. Parameter, result, and test method identification,
3. Sample-specific detection limits for each parameter, and
4. Results of laboratory control data (method blanks, spikes, and replicates as required).

12.3 DATA REPORTING

Data reporting is accomplished by using CLASS™. The data flow scheme for CLASS™ is presented in Figure 12-1. All client data and pertinent field information are entered into CLASS™ directly from the chain of custody sheets. A copy of this information is given to the Project Managers for verification to ensure that all pertinent information is available and correct. CLASS™ sorts all available samples for analyses for each parameter by due date, client ID, field group, etc. Weekly reports are generated by Laboratory Information Services and sent to each analytical department to notify them of samples that are due for analysis.

Figure 12-1 Flowchart of the CLASS™ Program

Sample Log-In	-Activate samples -Set permanent laboratory codes/identification -Store collection time and date -Store field data (from chain-of-custody)
Schedule Analyses	-List of samples available for each parameter sorted by due date
Input Results	-Reserve analysis -Input calibration, quality control, and sample data
Quality Control	-Calculate calibration curve, spike recovery, replicate and reference sample quality control
Calculate Results	-Calculate and store final sample concentrations
Intersample QC	-Check for data inconsistencies -Perform interparameter calculations
Reports	-Produce reports of sample data, quality control, and statistical analyses

Each analyst enters their analytical information into CLASS™ as a batch report. If applicable, the analysts enter standard curves (linear, quadratic, or logarithmic), method blanks, control spike data, as well as sample results into CLASS™ to create a batch. Final results are calculated according to the analytical methods specified in Section 5 of the CQAP. The analysts check all their data to ensure that all information is available and correct before signing the batch report. The analyst's Department Manager or designee then reviews the final batch report and signs it to verify that all data are accurate as reported. The batch is then finalized by the Department Manager or designee. Once a batch is finalized, the analyst cannot change the data. Any corrections are made by the Department Manager or designee (See Section 7). The Administrative staff generates and prepares, with data from CLASS™, the final report for the client. The Project Manager reviews the final reports for inconsistencies and completeness. An example Final Report is illustrated in Figure 12-2.

12.4 DATA STORAGE

A hard copy of all batch folders, supporting documents, and project files are filed chronologically by department in the secured centralized batch storage area. The newer batch folders are also stored chronologically by department in file cabinets located in Information Services Department. The batch folders include copies of sample preparation/digestion/extraction logbooks, copies of instrument logbooks, standard preparation logbook pages, sample arrival notices, CLASS™ batch reports, and raw data. The batch folders may be checked out for review by laboratory analysts, Laboratory Coordinators, or other laboratory personnel. In addition, any personnel checking out a batch folder from Laboratory Information Services is required to sign, date, indicate the batch numbers, and department numbers on the Document Control Logbook (Figure 12-3). When the laboratory analysts, Laboratory Coordinators, or other laboratory personnel are finished reviewing the batch folders, they are returned to Laboratory Information Services and the Document Control Logbook is signed and dated. At a minimum, all project files are kept for ten years.

The original laboratory logbooks and analysts logbooks are used until they are filled and are archived by the Department Manager.

All data stored in the CLASS™ database are backed up every weekday using high-density storage media. Tapes are stored in special files and are archived in a secured air-conditioned location (CLASS™ is discussed in further detail in Section 7).

Figure 12-2 Example Final Report

ESE Environmental Science & Engineering, Inc.
8901 North Industrial Road Peoria, IL 61615-1589
Phone (309) 692-4422 Lab Fax (309) 692-5232
An IEPA Contract Laboratory

TO:

ATTN:

REPORT DATE: 08-02-94
DATE RECEIVED: 07-25-94
PROJECT NUMBER:

ESE SAMPLE
SAMPLE DATE 07/22/94

DESCRIPTION	UNITS	GRAB WATER	METHOD NO.	DATE ANALYZED	ANALYST
METAL					
IRON	MG/L	13.2	200.7	07-27-94	ELZ
OTHER PARAMETERS					
PH	UNITS	7.38	150.1	07-26-94	AMH
TSS (RESIDUE, SUSP.)	MG/L	28	160.2	07-26-94	AMH
TDS (RESIDUE DISS,180 DEG)	MG/L	597	160.1	07-26-94	AMH
CHLORIDE	MG/L	87	4500C1B	08-01-94	KMC

Report Approved by: ____________________
Janel A. Woodin
Project Manager

Figure 12-3 Document Control Logbook

DOCUMENT CONTROL

PLEASE USE SEPARATE LINE FOR EACH BATCH CHECKED-OUT !

INITIALS	DATE/TIME OUT	DOCUMENT TYPE (e.g. Batch #, FG #, Files #, etc.)	DOC/ DEPT #	DATE/TIME RETURN	INITIALS	DEPT USE ONLY

13.0 CORRECTIVE ACTION

Corrective action is necessary when any measurement system fails to follow this CQAP. Items that may need corrective action range from a minor problem: a field team member failing to sign a field form, to a major problem: an analyst using an improper analytical method. For this reason, corrective action protocols are flexible.

13.1 ANALYTICAL

In general, items needing corrective action fall into three "correction" categories: short-term, long-term, and QC; each item requires different action.

13.1.1 SHORT-TERM CORRECTIVE ACTIONS

Short-term corrective actions consist of minor and major problems that are corrected immediately. Examples include failure to date or sign a standard form, incorrectly preserving sample, and errors in data entry. Corrective action is initiated by verbally calling attention to the problem followed by written notification in the logbook. All corrections will be documented in the project file.

13.1.2 LONG-TERM CORRECTIVE ACTIONS

Long-term corrective actions consist of minor and major problems that require a series of actions to resolve the problem. The actions taken are coordinated by the Laboratory Manager, Project Manager, or Laboratory QA/QC Coordinator; and a QA corrective action and routing form (Figure 13-1) is used to track the action. An example of this type of corrective action is as follows:

> Problem--A laboratory analyst fails to calibrate a pH meter prior to use.
>
> Corrective Action--The problem is identified by the person originating the corrective action, responsibility is assigned to an appropriate person (may be someone other than person failing to calibrate the instrument), re-training of the analysts on the use of the instrument is required, and the instrument is calibrated

Figure 13-1 CORRECTIVE ACTION REQUEST AND ROUTING FORM

ESE Peoria Laboratory

CA #

Department Identification:____________________________

1. Problem Identification

Name of Originator:____________________________ Date:____________

Nature of Problem: (Please include method where necessary.) ____________________

2. Required Action Determination

Responsibility Assigned:____________________________ Date:____________

Recommended Action:____________________________

3. Required Action Implementation

Responsibility Assigned:____________________________ Date:____________

4. Required Action Effectiveness Assurance

Responsibility Assigned:____________________________ Date:____________

Procedure to Assure Effectiveness:____________________________

5. QA/QC Acceptance

Corrective Action Status: _______Acceptable _______Unacceptable*

Signature of QA/QC Officer:____________________________ Date:____________

*If unacceptable, returned to:____________________________for revisions. Date:____________

*****PLEASE ATTACH ALL ASSOCIATED DOCUMENTATION.*****

prior to the next analysis. The Laboratory QA/QC Coordinator audits this process to assure that it is completed in an expeditious manner.

13.1.3 QUALITY CONTROL CORRECTIVE ACTION

Quality control corrective action consists of corrective action following a failure to meet QC criteria specified in this CQAP and the analytical methods. Actions taken consist of two types: those resolved within each analytical department and those resolved outside the department which requires a corrective action form. Examples outlining the differences between these two types of corrective action are as follows:

WITHIN DEPARTMENT ACTION

QC Failure	Department Action
Tuning results for GC/MS fail criteria in EPA Method 624	Analyst retunes instrument
Standard curve correlation coefficient is less than 0.995	Analyst investigates problem and reruns curve and samples
Sample response falls outside calibration curve	Analyst dilutes sample into range of curve

OUTSIDE DEPARTMENT ACTION

QC Failure	Department Action
Holding times are exceeded	Notify Project Manager, and Laboratory QA/QC Coordinator; resampling may be necessary; Project Manager contacts client

The corrective action procedures that are taken by the Peoria Laboratory following a failure to meet QC criteria specified in this CQAP and the analytical methods, except for CLP protocol, are summarized in Tables 13-1 through 13-5.

Outside Department corrective actions in the laboratory are documented and tracked using the Corrective Action Request and Routing Form (Figure 13-1).

Corrective actions are initiated for each measurement system (individual disciplines), by Department Managers, or other responsible individuals such as the Laboratory Project Managers or Laboratory Manager. On occasion, corrective actions are also initiated at the request of a client. The Laboratory QA/QC Coordinator is responsible for approving the corrective action for the client in the same fashion as if it had been initiated by laboratory personnel.

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Table 13-1. Summary of Corrective Action Procedures for Metals Analyzed by Graphite Furnace and Cold Vapor Atomic Absorption Spectroscopy

Quality Control	Acceptance Criteria	Corrective Action
Initial calibration verification standard (ICV)	± 10% of true value (GFAA) ± 20% of true value (CVAA)	Rerun standard, if still out of control, recalibrate instrument.
Calibration blank (ICB)	< RL (listed in Section 5)	Rerun the blank, if still out of control, reprocess and reanalyze the blank.
Calibration curve correlation coefficient	≥ 0.995	Rerun calibration standards, if still out of control, prepare new calibration standards and recalibrate the instrument or document why data are acceptable.
Calibration curve	Brackets all sample responses	Dilute and reanalyze within the calibration curve range or document why data are acceptable.
Continuing calibration verification standard (CCV)	± 20% of true value	Rerun standard, if still out of control, recalibrate instrument and reanalyze samples run since last acceptable CCV.
Method blank (MB)	< RL (listed in Section 5)	Determine the cause of the blank problem, redigest set, if necessary, or document why data are acceptable.

Table 13-1. Summary of Corrective Action Procedures for Metals Analyzed by Graphite Furnace and Cold Vapor Atomic Absorption Spectroscopy (Continued, Page 2 of 2)

Quality Control	Acceptance Criteria	Corrective Action
Standard matrix spike (SP)	See Section 5 for percent recovery control limits	Determine and correct problem, redigest and reanalyze samples, if necessary, or document why data are acceptable.
Sample matrix spike (SPM)	See Section 5 for percent recovery control limits	Determine and correct the problem, or document why data are acceptable.
Sample matrix spike duplicate	See Section 5 for RPD control limits	Determine and correct the problem, or document why data are acceptable.

Note: RPD = relative percent difference.
RL = reporting limit

Source: ESE.

Table 13-2. Summary of Corrective Action Procedures for Metals Analyzed by Inductively Coupled Plasma Emission Spectroscopy

Quality Control	Acceptance Criteria	Corrective Action
Initial calibration verification standard (ICV)	± 10% of true value	Rerun standard, if still out of control, recalibrate instrument.
Calibration blank (ICB)	< RL (listed in Section 5)	Rerun the blank, if still out of control, reprocess and reanalyze the blank.
Interference check standard (ICS)	± 20% of true value	Rerun standard, if still out of control, recalibrate instrument and reverify calibration.
Continuing calibration verification standard (CCV)	± 10% of true value	Rerun standard, if still out of control, recalibrate instrument and reanalyze all samples run since last acceptable CCV or document why data are acceptable.
Method blank (MB)	< RL (listed in Section 5)	Determine the cause of the blank problem; redigest samples if necessary or document why data are acceptable.

Table 13-2. Summary of Corrective Action Procedures for Metals Analyzed by Inductively Coupled Plasma Emission Spectroscopy (Continued, Page 2 of 2)

Quality Control	Acceptance Criteria	Corrective Action
Standard matrix spike (SP)	See Section 5 for percent recovery control limits	Determine and correct problem, redigest and reanalyze samples, if necessary, or document why data are acceptable.
Sample matrix spike (SPM)	See Section 5 for percent recovery control limits	Determine and correct problem, or document why data are acceptable.
Sample matrix spike duplicate	See Section 5 for RPD control limits	Determine and correct the problem, or document why data are acceptable.

Note: RL = reporting limit.
RPD = relative percent difference.

Source: ESE.

Table 13-3. Summary of Corrective Action Procedures for All Wet Chemistry Procedures

Quality Control	Acceptance Criteria	Corrective Action
Calibration curve correlation coefficient	$\geq$ 0.995	Rerun calibration standards if still out of control prepare new calibration standards and recalibrate the instrument, or document why data are acceptable.
Calibration curve	Brackets all sample responses	Dilute and reanalyze samples within the calibration curve range, or document why data are acceptable.
Calibration blank	< RL (listed in Section 5)	Rerun the blank, if still out of control, reprocess and reanalyze the blank.
Continuing calibration verification standard (CCV)	± 20% of true value	Rerun standard, if still out of control, recalibrate instrument and reanalyze samples run since last acceptable CCV or document why data are acceptable.
Method blank (MB)	< RL (listed in Section 5)	Determine the cause of the blank problem, reanalyze samples, if necessary, or document why data are acceptable.
Sample replicate (RP)*	See Section 5 for RPD control limits	Determine and correct the problem, reanalyze samples, if necessary, or document why data are acceptable.

Table 13-3.
Summary of Corrective Action Procedures for All Wet Chemistry Procedures (Continued, Page 2 of 2)

Quality Control	Acceptance Criteria	Corrective Action
Standard matrix spike (SP)	See Section 5 for percent recovery control limits	Determine and correct problem, reanalyze samples if necessary or document why data are acceptable.
Sample matrix spike (SPM)	See Section 5 for percent recovery control limits	Determine and correct the problem, or document why the data are acceptable.
Sample matrix spike duplicate	See Section 5 for RPD control limits	Determine and correct the problem, or document why the data are acceptable.

Note: RL = reporting limit.
RPD = replicate percent difference.

*Sample replicate is only required for miscellaneous inorganic parameters including residues, pH, specific conductivity, turbidity, dissolved oxygen, and % moisture analyses.

Source: ESE.

Table 13-4. **Summary of Corrective Action Procedures for Organics Analyzed by Gas Chromatography and High Pressure Liquid Chromatography**

Quality Control	Acceptance Criteria	Corrective Action
Calibration curve correlation coefficient	≥ 0.995	Rerun calibration standards, if still out of control, prepare new calibration standards and recalibrate the instrument, or document why the data are acceptable.
Calibration curve	Brackets all sample responses	Dilute and reanalyze samples within the calibration curve range, or document why data are acceptable.
Continuing calibration standard (CCS)	± 15% of standard initial response for GC EPA SW-846 and ± 10% for GC EPA 600s ± 10% of standard initial response for HPLC. Drinking water ± 20%	Rerun standard, if still out of control, recalibrate instrument and reanalyze samples when last CCS is acceptable, or document why data are acceptable.
Method blank (MB)	< than RL for organics (listed in Section 5)	Determine and correct cause of the blank problem, reanalyze the samples, if necessary, or document why data are acceptable.
Sample matrix spike (SPM)	See Section 5 for percent recovery control limits	Determine and correct the problem, or document why the data are acceptable.

Table 13-4.
Summary of Corrective Action Procedures for Organics Analyzed by Gas Chromatography and High Pressure Liquid Chromatography (Continued, Page 2 of 2)

Quality Control	Acceptance Criteria	Corrective Action
Sample matrix spike duplicate	See Section 5 for RPD control limits	Determine and correct the problem, or document why the data are acceptable.
Standard matrix spike (SP)	See Section 5 for percent recovery control limits	Determine and correct the problem, reanalyze samples if necessary or document why the data are acceptable.
Surrogates* (SUR)	See Section 5 for percent recovery control limits	Reanalyze samples with surrogates outside criteria or document why data are acceptable.

Note: RL = reporting limit.
GC = gas chromatography.
HPLC = high pressure liquid chromatography.
RPD = relative percent difference.

*Surrogate/surrogates will only be spiked in samples if specified by the method.

Source: ESE.

Table 13-5.
Summary of Corrective Action Procedures for Organics by Gas Chromatography/Mass Spectrometry

Quality Control	Acceptance Criteria	Corrective Action
DFTPP or BFB instrument tuning	See Section 9 for tuning criteria	Retune instrument until within criteria.
Initial calibration standards	See Section 9 for calibration criteria	Rerun calibration standards, if still out of criteria, prepare new calibration standards and rerun standards.
One-point daily calibration	See Section 9 for calibration criteria	Rerun standard, if still out of control, rerun calibration curve, or document why data are acceptable.
Method blank (MB)	< two times the RL (listed in Section 5) for semivolatile organics	Evaluate the impact of the presence of any target analytes in the method blank, the presence of low concentrations of phthalate are acceptable. Reextract and reanalyze samples if presence of target analytes are unacceptable or document why data are acceptable.

Table 13-5.
Summary of Corrective Action Procedures for Organics by Gas Chromatography/Mass Spectrometry (Continued, Page 2 of 3)

Quality Control	Acceptance Criteria	Corrective Action
Method blank (MB)	No greater than 5 times the RL for methylene chloride, acetone, toluene, and xylene for volatile organics. All other analytes ≤ RL (listed in Section 5)	Reanalyze another MB. If second MB exceeds criteria, clean and recalibrate the analytical system or document why data are acceptable.
Surrogate (SUR)	See Section 5 for percent recovery control limits	If surrogates in the MB or SP are within limits, qualify the data. Reanalyze samples with surrogates outside criteria or document why data are acceptable.
Standard matrix spike (SP)	See Section 5 for percent recovery control limits	Determine and correct the problem, reanalyze samples if necessary, or document why data are acceptable.

Table 13-5.
Summary of Corrective Action Procedures for Organics by Gas Chromatography/Mass Spectrometry (Continued, Page 3 of 3)

Quality Control	Acceptance Criteria	Corrective Action
Sample matrix spike (SPM)	See Section 5 for percent recovery control limits	Determine and correct the problem, or document why the data are acceptable.
Sample matrix spike duplicate	See Section 5 for RPD control limits	Determine and correct the problem, or document why the data are acceptable.

Note: RL = reporting limit.
RPD = relative percent difference.

Source: ESE.

13.2 EXTERNAL SOURCES

Corrective actions are also initiated from external sources. This includes performance sample results, split samples, audits (for example, on-site or field by IEPA, FDEP, Army Corps of Engineers, etc.), and data validation/review. Corrective actions recommended by agencies such as USEPA, FDEP, IEPA, etc. are prioritized, promptly acted on, and overseen by the Project Manager or Laboratory QA/QC Coordinator. Actions taken to resolve the problem are documented and kept by the Laboratory QA/QC Coordinator.

14.0 PERFORMANCE AND SYSTEM AUDITS AND PERSONNEL TRAINING

14.1 INTRODUCTION

Two types of audit procedures are used to assess and document performance of laboratory staff: system audits and performance audits. These are performed at frequent intervals by the Laboratory QA/QC staff. These audits form one of the bases for corrective action requirements and constitute a permanent record of the conformance of measurement systems to QA requirements.

14.2 SYSTEM AUDITS

System audits are inspections of training status, records, QC data, calibrations, and conformance to SOPs without the analysis of check samples. System audits are performed quarterly by the Laboratory QA/QC Coordinator or appointed individual.

The system audit protocol for the laboratory is summarized as follows:

1. Laboratory Operations - The Laboratory QA/QC Coordinator or appointed individual will perform the quarterly laboratory system audit using the checklist in Figures 14-1 through 14-4. The items to be reviewed are:

   a. Parameter and/or laboratory notebooks;
   b. Instrument logbooks;
   c. Sample log-in, dispensing, and labeling for analysis;
   d. QC criteria update for spike recoveries; and
   e. Verify that deficiencies in the last audit were corrected.

   In addition, the Laboratory QA/QC Coordinator monitors methods randomly to assure adherence to approved analytical methods.

2. Final Reports - As a normal work process, the Project Manager reviews all final reports and deliverables before they are sent to the client.

Figure 14-1 Checklist For Coldrooms, Freezers and Sample Storage Areas

Coldrooms, Freezers and Sample Storage

Department:________________________________

ITEM	YES	NO*	COMMENTS
1. Is the work area clean and organized?			
2. Are SOPs available for receipt, storage, and tracking of samples?			
3. Are there findings in this department from last quarter's lab audit? If yes, list below (or attach a separate sheet) and verify that they have been corrected.			
4. Are documentation errors corrected properly (one line drawn through error, date, error code/explanation, and initials)?			
5. Are the Sample Tracking forms properly filled out?			
6. Is the Sample Location report updated on a regular basis and placed next to the door of each storage area?			
7. Are all storage areas secured at all times?			
8. Are the temperature logs for the coldrooms and freezers filled out completely and corrections made properly? Are appropriate corrective actions taken for all out-of-control readings?			
9. Is a condensed SOP for check-in/check-out log filled out completely?			
10. Is the Sample Check-In/Check-Out log filled out completely?			
11. Is proper documentation available for tracking the disposal of samples?			
Additional Comments:			

*For all "No" answers, include all information necessary to trace audit finding (e.g., Rm. #, logbook #, Page #, instrument #, etc.)

Figure 14-2 Checklists For Sample Receiving and Hood Maintenance

Sample Receiving

Department:____________________________________

ITEM	YES	NO*	COMMENTS
1. Is the work area clean and organized?			
2. Are SOPs available for receipt, log-in and transfer of samples to storage areas?			
3. Are there findings in this department from last quarter's lab audit? If yes, list below (or attach a separate sheet) and verify that they have been corrected.			
4. Are documentation errors corrected properly (one line drawn through error, date, error code/explanation, and initials?			
5. Is the Sample Custodian filling out all required information on the chain of custody (COC) form (cooler temp., seals intact? etc.)?			
6. Are the Sample Chest Custody Forms filled out completely?			
7. Is the Sample Custodian completely filling out the Cold Room Sample Arrival logbook?			
8. Is the Sample Custodian auditing 10% of all samples (except VOA samples) to verify that samples are properly preserved? Is documentation available?			
9. Are samples labelled properly?			
Additional Comments:			

Hood Maintenance

Department:____________________________________

ITEM	YES	NO*	Comments
1. Have fume hoods been calibrated within the last year? Are they labelled as to when last tested?			

*For all "No" answers, include all information necessary to trace audit finding (e.g., Rm#, logbook #, page #, instrument #, etc.)

Figure 14-3 **Checklist For Sample Kit Prep Area**

Sample Kit Prep Area

Department: ______________________

ITEM	YES	NO*	COMMENTS
1. Is the work area clean and organized?			
2. Are SOPs available?			
3. Are there findings in this department from last quarter's lab audit? If yes, list below (or attach a separate sheet) and verify that they have been corrected.			
4. Are documentation errors corrected properly (one line drawn through error, date, error code/explanation, and initials)?			
5. Are all preservatives labelled properly?			
6. Is the sample Kit Prep & Shipping Request Form filled out completely?			
7. For coolers picked up[by field personnel, is the appropriate information documented in the Kit Pick-up log? Is the Kit Pick-up log signed by both kit prep and field personnel?			
8. For coolers shipped to the field, is the appropriate information documented in the Shipping receipt (ice chest check out) log?			
9. Is a copy of the Shipping Receipt (ice chest check out) form attached to the Kit Prep & Shipping Request form?			
Additional Comments:			

*For all "No" answers, include all information necessary to trace audit finding (e.g., Rm#, logbook #, Page #, instrument #, etc.)

Figure 14-4
Checklist For Laboratory Area Responsibilities and Glassware Washing Procedures

Laboratory Area Responsibilities

Department: ______________________________

ITEM	YES	NO*	COMMENTS
1. Have fume hoods been calibrated within the last year? Are they labelled as to when last tested?			
2. Are refrigerator/freezer temperature logs filled out completely and corrections made properly? Are temperatures taken daily, except weekend days? Are appropriate corrective actions taken for any out-of-control readings?			
3. Are the balance calibration logs filled out completely and corrections made properly? Are balances calibrated daily, except weekend days, for analytical balances and weekly for top loading balances? Are appropriate corrective actions taken for any out-of-control readings?			
4. Is the balance manufacturer's maintenance done annually?			
5. Are documentation errors for these logbooks corrected properly (one line drawn through error, date, error code/explanation, and initials)?			
Additional Comments:			

Glassware Washing Procedures

Department: ______________________________

Item	Yes	No*	Comments
1. Is the work area clean and organized?			
2. Are SOPs available?			
3. Are there findings in this department from last quarter's lab audit? If yes, list below (or attach a separate sheet) and verify that they have been corrected.			
4. Are documentation errors corrected properly (one line drawn through error, date, error code/explanation, and initials)?			
5. Is clean glassware stored so as to avoid contamination?			
6. Are the Glassware Washing Request Forms filled out completely and signed and dated?			
7. Are properly labelled waste containers available?			
8. Is the deionized water system checked regularly to verify that it meets requirements?			
Additional Comments:			

*For all "No" answers, include all information necessary to trace audit finding (e.g., Rm#, logbook #, page #, instrument #, etc.)

Figure 14-5 **Checklist For Sample Preparation Areas**

Sample Preparation Areas

Department:

ITEM	YES	NO*	COMMENTS
1. Is the work area clean and organized?			
2. Are SOPs available for receipt, storage and tracking of samples?			
3. Are there findings in this department from last quarter's lab audit? If yes, list below (or attach a separate sheet) and verify that they have been corrected.			
4. Are documentation errors corrected properly (one line drawn through error, date, error code/explanation, and initials)?			
5. Are samples and standards stored separately to avoid contamination?			
6. Are spike solutions, surrogate solutions, (Org. only) and reagents labelled clearly and appropriately (including plastic squeeze bottles)?			
7. Are there expired standards/reagents in the laboratory? Are they clearly labelled as "expired" or "for qualitative use only"?			
8. Is glassware stored so as to avoid contamination?			
9. Do all log books have control numbers?			
10. Are sample preparation logs completely filled out, including preparer and reviewer signatures?			
11. Are automatic pipettes and syringes calibrated each day of use? (Inorganics Division only) Are all water bath thermometers in use calibrated against a NIST thermometer? (Organic Division) Are the calibrations documented in the appropriate logbooks?			
12. Are instrument run logs made properly (e.g., microwave, GPC)?			
13. Are instrument maintenance logs filled out completely and corrections made properly?			
14. Are extracts (sample vials) labelled properly?			
15. Are sample extract/digest chain of custody logs filled out completely and corrections made properly?			
16. Are properly labeled waste containers available?			

Comments:

*For all "No" answers, include all information necessary to trace audit finding (e.g., Rm.#, logbook #, page #, instrument #, etc.)

Figure 14-6 **Checklist For Sample Analysis Area**

Sample Analysis Areas

Department:________________________________

ITEM	YES	NO*	COMMENTS
1. Is the work area clean and organized?			
2. Are SOPs available?			
3. Are there findings in this department from last quarter's lab audit? If yes, list below (or attach a separate sheet) and verify that they have been corrected.			
4. Are documentation errors corrected properly (one line drawn through error, date, error code/explanation, and initials)?			
5. Are samples and standards stored separately to avoid contamination?			
6. Are spike solutions, surrogate solutions (Org. only), calibration standards and reagents labelled clearly and appropriately (including plastic squeeze bottles)?			
7. Is glassware stored so as to avoid contamination?			
8. Do all logbooks have control numbers?			
9. Are standard and reagent prep. logbooks filled out completely and corrections made properly? Are lot numbers of neat standards recorded?			
10. Are instrument calibration checks performed prior to analysis? (Mandatory for Radiochemistry, only)			
11. Are instrument run logs filled out completely and corrections made properly?			
12. Are instrument maintenance logs filled out completely and corrections made properly.			
13. Are samples (analysis vials) labelled properly?			
14. Are sample chain-of-custody (COC) logs (VOA samples) or sample extract/digest COC logs filled out completely and corrections made properly?			
15. Are properly labeled waste containers available?			
Additional Comments:			

*For all "No" answers, include all information necessary to trace audit finding (e.g., Rm.#, logbook #, page #, instrument #, etc.)

Figure 14-7 **Checklist For Information Services**

Information Services

Department: ______________________

ITEM	YES	NO*	COMMENTS
1. Is the work area organized?			
2. Are appropriate SOPs available?			
3. Are there findings in this department from last quarter's lab audit? If yes, list below (or attach a separate sheet) and verify that they have been corrected.			
4. Are documentation errors corrected properly (one line drawn through error, date, error code/explanation, and initials)?			
5. Are the Chain-of Custody Forms properly filed and readily accessible?			
6. Are the filing cabinets where data are stored kept locked?			
7. Are batch folders readily accessible?			
8. Is the Document Control Logbook filled out completely?			
9. Are the appropriate approval forms and signatures maintained for changes to finalized data batches or CLASS™ STORET files?			
Additional Comments:			

*For all "No" answers, include all information necessary to trace audit finding (e.g., Rm.#, logbook #, page #, instrument #, etc.)

The Peoria Laboratory is audited by external sources, such as states and federal agencies, as well as internal sources, such as the Laboratory QA/QC staff. External audits are conducted to verify compliance with rules, regulations, or criteria for certification. External audits are conducted with a high degree of formality. The Peoria Laboratory is externally audited regularly by the following agencies:

1. State of Illinois Environmental Protection Agency,
2. State of New Jersey Department of Environmental Protection and Energy,
3. State of California Department of Health Services,
4. State of New Hampshire Department of Environmental Services,
5. State of Wisconsin Department of Natural Resources,
6. State of North Carolina Department of Environment, Health, and Natural Resources, and
7. United States Army Corps of Engineers.

ESE submits to periodic external audits after notification and scheduling by the Laboratory QA/QC Coordinator and Laboratory Manager.

14.3 PERFORMANCE AUDITS

Performance audits are inspections of the on-going quality program in the laboratory focusing on the evaluation of the accuracy of all laboratory data. Performance audits are conducted quarterly by the Laboratory QA/QC Coordinator or appointed individual.

The results of interlaboratory studies are evaluated by the Laboratory QA/QC Coordinator as part of the performance audits. This type of evaluation is performed at least quarterly. ESE participates in the following proficiency programs:

1. USEPA Water Pollution and Water Supply proficiency programs,
2. USEPA National Pollutant Discharge Elimination System (NPDES) DMR-QA proficiency program,

3. American Industrial Hygiene Association (AIHA), Environmental Lead Proficiency Analytical Testing (ELPAT) program,
4. State of Wisconsin, State Laboratory of Hygiene,
5. U.S. Army Corps of Engineers, and
6. Analytical Standards, Inc., Environmental Performance Audit (EPA)™ program.

Besides participation in several proficiency programs, the ESE Peoria Laboratory is currently certified by numerous state and regulatory agencies which require verification of laboratory's proficiency on an annual basis. The following licenses, accreditations, certifications and validations are held by the Peoria Laboratory:

1. State of California Department of Health Services,
2. State of Connecticut Department of Health Services,
3. State of Illinois Environmental Protection Agency,
4. State of Illinois Contract Laboratory Program,
5. State of Illinois Department of Public Health,
6. State of Iowa Department of Natural Resources,
7. State of Kansas Department of Health and Environment,
8. State of Kentucky Department For Environmental Protection,
9. State of New Hampshire Department of Environmental Services,
10. State of New Jersey Department of Environmental Protection and Energy,
11. State of North Carolina Department of Health, Environment, and Natural Resources,
12. State of Oklahoma Water Resources Board,
13. State of Pennsylvania Department of Environmental Resources,
14. State of Tennessee Department of Environment and Conservation,
15. State of Washington Department of Ecology,
16. State of Wisconsin Department of Natural Resources, and
17. United States Army Corps of Engineers.

In addition to reviewing performance evaluation program results, ESE Peoria performs quarterly data audits. An analytical batch is audited randomly using the Data Validation Checklist, Figure 14-8. All audit findings are kept on file in the QA/QC office. On a daily basis, peer review of all deliverable reports and data are performed by technically qualified individuals from each major discipline represented in the deliverable.

14.4 PERSONNEL TRAINING

The Peoria Laboratory personnel are trained in health and safety, QA/QC procedures, analytical methods, and the laboratory data management system. New personnel are trained prior to performing any actual laboratory work. Laboratory personnel are also required to attend the health and safety and laboratory QA/QC procedures refresher courses offered yearly. Training that each laboratory personnel receives is documented by the Laboratory Department Manager in the personnel's training records maintained by the Laboratory QA/QC staff.

Figure 14.8 DATA REVIEW/VALIDATION CHECKLIST

ESE Batch ______________ Dept. ______________ Method ____

Project Name ______________ Field Group ______________

1. BATCH PRINTOUT	YES	NO	N/A	COMMENTS
A. HEADER PAGE				
Are the digestion/extraction and analysis dates consistent with those in the raw data?				
Are the sample volumes/weights consistent with those on the raw data?				
Are the correct units specified?				
For dry weight parameters, is there a % moisture of ≤ 0.05 listed for any data sample? If so, verify that % moisture was performed on this sample.				
Is the appropriate QC type specified?				
Is the method blank correction appropriate to the method/project?				
Are the required QC samples (MB, SP, SPM1, SPM2, RP, SUR) analyzed at the required frequency?				
Are frequency criteria for ICV/CCB/CCV/CCS met?				
B. RAW DATA				
Is the MB free of contamination? If not, is an explanation provided?				
Are the raw data present and identified properly? Are dilution factors entered correctly?				
Are the units specified correctly?				
Have the QC samples been reported using the sample calibration curve as the data samples?				

Figure 14.8 DATA REVIEW/VALIDATION CHECKLIST (Continued, Page 2 of 4)

1. BATCH PRINTOUT (Cont'd)	YES	NO	N/A	COMMENTS
C. Calibration and QC Summary				
Is the required number of standards for the method analyzed? If not, is an explanation provided?				
Is the correlation coefficient of the curve ≥0.995?				
Is the predictability for the curve acceptable for the data reported? (e.g., Is the predictability good at the detection limit for all "less than" samples?)				
Are the standards uploaded/entered correctly in CLASS™?				
If reanalysis or dilutions are performed on a different day, is a new standard curve analyzed?				
Are detection limits reported correctly?				
Do the QC samples (MB, SP, SPM1, SPM2, RP, SUR) meet acceptance criteria (accuracy/precision)?				
If not, are explanations provided in the data batch?				
Do the calibration QC samples (ICV/CCB/CCV/CCS) meet acceptance criteria?				
If not, are explanations provided in the data batch?				
D. SAMPLE DATA				
Are reported concentrations within the calibration range?				
If not, are samples diluted within the calibration range?				
Are the reported values consistent with the required units?				

Figure 14.8 DATA REVIEW/VALIDATION CHECKLIST (Continued, Page 3 of 4)

1. BATCH PRINTOUT (Cont'd)	YES	NO	N/A	COMMENTS
Is an example calculation provided in the batch?				
E. CHECKLISTS				
Was extraction/digestion HT met?				
Was analysis HT met?				
Were dilutions performed within HT?				
Are any "No" answers in the Computer QC and Manual checklists provided with explanations/corrective actions?				
Is the Manual Checklist in the data batch being filled out completely?				
Are all comments dated and initialed?				
Are the appropriate signatures present on the checklist?				
2. DOCUMENTATION				
Is sample prep, extraction, or digestion log present?				
Are standards, spikes, and reagents prep logs present?				
Are all necessary forms present/complete?				
Are all notebook/log pages legible, signed, and dated?				
Are all documentation errors made properly (one line drawn, dated, initialed, and error code/explanation provided)?				
Are all notebook/log pages filled out completely?				
Are instrument run logs present, legible, and completely filled out?				

Figure 14.8 DATA REVIEW/VALIDATION CHECKLIST (Continued, Page 4 of 4)

3. RAW DATA (Chromatograms, worksheets, upload summary files, strip charts, etc.)	YES	NO	N/A	COMMENTS
Are the raw data present and identified properly? (Analyst name, analysis date, instrument conditions, peaks labelled, dilution, etc.)				
Is justification provided for non-used data?				
Has second confirmation been performed for all positive identification made in the primary analysis? (For GC and HPLC only) Have they been performed within HT?				
Is the 12/24-hour tuning criteria met? (For GC/MS only)				
Have SPCC, RRF, and CCC criteria been met? (For GC/MS only)				

	YES	NO	N/A	COMMENTS
4. CALCULATION VERIFICATION				
Randomly select one or more values and trace back the calculations to the raw data. Do the numbers agree?				
Sample No: Value Calculated: Value Reported in Batch:				
ADDITIONAL COMMENTS:				

Auditor: ____________________ Date Review Completed: ____________________

15.0 QUALITY ASSURANCE REPORTS

Project Quality Assurance reports are either internal or external in nature. Upon request, a Project QA report is written upon completion of the project or immediately upon the discovery of a problem requiring corrective action. The Inorganic and Organic Operations Manager is responsible for compiling the QA information provided by the Department Managers and submitting the complete report to the client/agency. Activities and actions to be reported will include:

1. An assessment of the project's status in relation to the progress of proposed time table;
2. Results of ongoing performance and system audits (Results of other performance and system audits are reported to management quarterly by the Laboratory QA/QC Coordinator);
3. Assessment of measurement data accuracy, precision, and method detection limits; and
4. Data quality review and significant QA problems with proposed corrective action procedures.

The Department Managers, Project Managers, and Laboratory QA/QC Coordinator are informed of the contents of the final Project QA report by the Inorganic and/or Organic Operations Manager through review of the final report.

16.0 PERSONNEL SUMMARY AND RESUMES

Table 16-1 lists the titles and positions of all laboratory personnel currently employed at the ESE Peoria Laboratory.

ESE PEORIA ANALYTICAL LABORATORY

Personnel and QA Officer Summary

TITLE	NAME	DEGREE/YEAR BACKGROUND	YEARS EXPERIENCE
Laboratory Manager	Kim D. Johnson	B.S., 1989, Business Management, Laboratory Director	15
Operations Manager--Inorganics	Ty R. Woodin	B.S., 1985, Chemistry, Operations Management	8
Customer Services Manager	Barb G. Raya-Flash	B.S., 1976, Environmental Biology, Project Management	17
Laboratory Project Manager	Vickie M. Wynkoop	B.S., 1978, Biology, Project Management	11
Laboratory Project Manager	Janel A. Woodin	B.S., 1987, Chemistry, Project Management	6
Laboratory Project Coordinator	Jim T. McQuellon	B.S., 1984, Chemistry, Project Management	9
Laboratory Project Coordinator	Dorothy W. Rothert	B.S., 1962, Chemistry, Project Coordination	9
Department Manager--Laboratory Information Services	Dean J. Huhmann	B.S., 1986, Management Information Systems LIMS Management	10
Operations Manager--Organics	Barbara J. Beard	B.S., 1976, Biology/Chemistry Operations Management	18
Laboratory QA/QC Coordinator	Lettie E. Schmitt	B.S., 1992, Biology/Chemistry QA/QC	4
Sample Custodian	Alan R. Hogan	A.S., 1981, Liberal Arts, Sample Receiving	12
Staff Lab Scientist	Skip D. Reining	B.S., 1985, Admin. Office Mgt., Sample Receiving	2
Senior Staff Lab Scientist	Sandi L. Lawson	B.S., 1985, Geology, Chromatography	8

TITLE	NAME	DEGREE/YEAR BACKGROUND	YEARS EXPERIENCE
Staff Lab Scientist	Rick D. Urish	B.S., 1985, Agriculture, Chromatography	4
Staff Lab Scientist	Rachel A. Harris	B.S., 1993, Agricultural Science Chromatography	2
Staff Lab Scientist	Frank W. Mueller	B.A., 1990, Chemistry Chromatography	3
Staff Lab Scientist	Judy A. Zosky	High School Chromatography	3
Staff Lab Scientist	Brent A. Donalson	B.S., 1991, Chemistry Chromatography	2
Staff Lab Scientist	William T. Franey	B.S., 1991, Agriculture Chromatography	2
Staff Lab Scientist	Angela M. Whiting	B.S., 1992, Environmental Health Chromatography	1
Staff Lab Scientist	Lynn E. Isbell	M.S., 1990, Chemistry Chromatography	7
Staff Lab Scientist	Tom E. Westrick	B.A., 1993, Chemistry Chromatography	1
Senior Staff Lab Scientist	Kevin M. Cranford	B.S., 1984, Science Inorganic Chemistry	5
Staff Lab Scientist	Chris J. Fisher	B.S., 1987, Biology Inorganic Chemistry	6
Staff Lab Scientist	Frances T. Jacobs	High School Inorganic Chemistry	9
Department Manager–Sample Preparation	Kirby R. Feldmann	B.S., 1984, Biological Sciences Organic Extraction	9
Staff Lab Scientist	Todd J. Peterson	B.S., 1991, Chemistry Organic Extraction	3
Staff Lab Scientist	Kristina M. Ericcson	B.S., 1993, Microbiology/ Chemistry, Organic Extraction	1
Staff Lab Scientist	Michele R. Larsen	B.S., 1992, Chemistry Chromatography	2
Department Manager–GC/MS	Sandra K. Boucher	B.S., 1974, Biological Science Mass Spectrometry	5

TITLE	NAME	DEGREE/YEAR BACKGROUND	YEARS EXPERIENCE
Staff Lab Scientist	Leslie D. Miller	B.S., 1987, Education B.A., 1990, Chemistry *Mass Spectrometry*	4
Staff Lab Scientist	Doug A. Hafley	B.S., 1990, Chemistry Mass Spectrometry	8
Staff Lab Scientist	Kent A. Fisher	B.S., 1990, Environmental Biology Mass Spectrometry	3
Staff Lab Scientist	Annette M. Heinie	B.S., 1990, Chemistry Mass Spectrometry	3
Staff Lab Scientist	Melissa J. Lentz	B.A., 1990, Chemistry Mass Spectrometry	4
Staff Lab Scientist	Rosey M. Murton	B.S., 1992, Biology Mass Spectrometry	1
Department Manager–Atomic Spectroscopy and Water Quality	William B. Gray	B.S., 1985, Biology Spectroscopy	7
Senior Staff Lab Scientist	Gregory R. St. Aubin	B.S., 1988, Agriculture/Agronomy Spectroscopy	5
Staff Lab Scientist	Deborah A. Blahnik	LPN, 1973 Spectroscopy	8
Senior Staff Lab Scientist	Ellen L. Zuck	B.S., 1988, Biology Spectroscopy	5
Staff Lab Scientist	John E. Lebak	High School Spectroscopy	2
Administrative Assistant–Financial	Sandra D. Heine	High School Administration	28
Administrative Assistant	Joan M. VanLoo	High School Administration	15
Administrative Assistant–Purchasing	Sandra M. Coursey	High School Administration	16
Administrative Assistant	Ann M. Dwyer	B.S., 1989, Business Administration	7
Secretary	Amy K. Smith	High School Administration	5

TITLE	NAME	DEGREE/YEAR BACKGROUND	YEARS EXPERIENCE
Laboratory Information Services	Jane A. Strevils	High School Document Control	8

LES:Labpersonnel.Summary
10/01/94

ANALYSIS OF VOLATILE ORGANIC COMPOUNDS
IN AMBIENT AIR SAMPLES BY GC/MS

(ESE Denver)

I. SUMMARY

A. ANALYTES

This method is applicable to the Class 1A analysis of the following compounds by Gas Chromatography/Mass Spectrometry (GC/MS):

Analyte

Benzene
Bicycloheptadiene (BCHPD)
Carbon Tetrachloride
Chlorobenzene
Chloroform
1,1-Dichloroethane
1,2-Dichloroethane
trans-1,2-Dichloroethene
Dicyclopentadiene (DCPD)
Dimethyldisulfide (DMDS)
Ethylbenzene
Methylisobutylketone (MIBK)
Tetrachloroethene
Toluene
1,1,1-Trichloroethane
1,1,2-Trichloroethane
Trichloroethene
Total Xylenes (*o,m,p*)

SURROGATES

Toluene-d8
Bromofluorobenzene

In addition, this method is applicable to the screening of additional volatile organic compounds which can be captured on Tenax® [poly(2,6-Diphenylphenylene oxide)] and determined by thermal desorption GC/MS techniques. Compounds which can be determined by this method are non polar organic compounds having boiling points in the range of approximately 40° - 200°C.

B. MATRIX

This method is applicable to organic compounds in ambient air that can be trapped on Tenax® (2,6-Diphenylphenylene oxide) cartridges.

C. GENERAL METHOD

Ambient air is drawn through two sorbent cartridges connected in series, the front cartridge containing Tenax®, and the second trap containing a mixture of Tenax® and activated carbon. The cartridges are then transported to the laboratory and analyzed for volatile organic compounds by GC/MS.

The Tenax trap, or the Tenax-carbon trap, is analyzed by placing it in a heated chamber (225°C) and purging the trap with helium for 11 min at a flow rate of 30 ml/min. The trap effluent is delivered into a Tekmar liquid concentrator where the sample analytes are re-concentrated. The Tekmar concentrator tube is then heated to 185°C and the volatile organic compounds transferred onto a Megabore DB-624 column for temperature programmed gas chromatography (GC) separation. Compounds from the GC column are detected and quantified by low-resolution mass spectroscopy. Quantitation is performed using internal standard techniques.

II. APPLICATION

A. TESTED CONCENTRATION RANGE

The tested concentration ranges in total micrograms (ug) for all the compounds are as follows:

Benzene	0.012 - 1.0
Bicycloheptadiene (BCHPD)	0.012 - 1.0
Carbon Tetrachloride	0.012 - 1.0
Chlorobenzene	0.012 - 1.0
Chloroform	0.012 - 1.0
1,1-Dichloroethane	0.012 - 1.0
1,2-Dichloroethane	0.012 - 1.0
Dicyclopentadiene (DCPD)	0.012 - 1.0
trans-1,2-Dichloroethene	0.012 - 1.0
Dimethyldisulfide (DMDS)	0.012 - 1.0
Ethylbenzene	0.012 - 1.0
Methylene Chloride	0.012 - 1.0
Methylisobutylketone (MIBK)	0.012 - 1.0
Tetrachloroethene	0.012 - 1.0
Toluene	0.012 - 1.0
1,1,1-Trichloroethane	0.012 - 1.0
1,1,2-Trichloroethane	0.012 - 1.0
Trichloroethene	0.012 - 1.0
Total Xylenes	0.036 - 3.0

Surrogate Compounds for GC/MS Method

Toluene-d8	0.012 - 1.0
Bromofluorobenzene	0.012 - 1.0

Quantitation ions and the corresponding internal standards for each analyte are listed in Table II.A.1.

Note: Methylene chloride, dibromochloropropane (DBCP) and 1,2-dichloroethane-d4 (as a surrogate) were attempted for this certification at the tested concentrations listed above. Due to poor accuracy and precision values generated for these three compounds, the certification data was unacceptable, and they are not included as certified compounds for this method.

Table II.A.1. Target Analytes: Internal Standards and Quantitation Ions

Analyte	Quantitation Ion	Internal Standard*
Benzene	78	1
BCHPD	91	2
Carbon Tetrachloride	117	1
Chlorobenzene	112	3
Chloroform	83	1
1,1-Dichloroethane	63	1
1,2-Dichloroethane	62	1
DCPD	66	3
trans-1,2-Dichloroethene	96	1
DMDS	94	2
Ethylbenzene	106	3
MIBK	43	2
Tetrachloroethene	164	2
Toluene	92	2
1,1,1-Trichloroethane	97	1
1,1,2-Trichloroethane	87	2
Trichloroethene	130	2
o-Xylene	91	3
p-Xylene and *m*-Xylene	91	3
Surrogate Compounds for GC/MS Method		
Toluene-d8	98	2
Bromofluorobenzene	95	3

* LEGEND FOR INTERNAL STANDARDS

Code	Internal Standard	Quantitation Ion
1	Bromochloromethane	128
2	1,4-Difluorobenzene	114
3	Chlorobenzene-d5	117

B. SENSITIVITY

The extracted ion current area count responses at the certified reporting limits (CRL) are:

Analyte	Area Count at the CRL*
Benzene	16300
BCHPD	9450
Carbon Tetrachloride	5900
Chlorobenzene	10900
Chloroform	6420
1,1-Dichloroethane	6980
1,2-Dichloroethane	5930
trans-1,2-Dichloroethene	3250
DCPD	122000
DMDS	16900
Ethylbenzene	9140
MIBK	65000
Tetrachloroethene	3870
Toluene	8940
1,1,1-Trichloroethane	9390
1,1,2-Trichloroethane	8500
Trichloroethene	3960
Total Xylenes	129000

*Note: These are area counts derived (by ratio) from the analytical standards (from the first day's certification) that are closest to the method CRL.

C. CERTIFIED REPORTING LIMITS

The CRL's calculated according to the USATHAMA reporting limit programs are:

Analyte	CRL (ug)	Upper Certified Range (ug)	Accuracy
Benzene	0.016	1.0	0.980
BCHPD	0.012	1.0	0.930
Carbon Tetrachloride	0.018	1.0	1.06
Chlorobenzene	0.013	1.0	0.950
Chloroform	0.012	1.0	0.981
1,1-Dichloroethane	0.012	1.0	0.961
1,2-Dichloroethane	0.012	1.0	0.992
trans-1,2-Dichloroethene	0.012	1.0	0.912
DCPD	0.089	1.0	1.14
DMDS	0.047	1.0	1.08
Ethylbenzene	0.032	1.0	0.990
MIBK	0.165	0.6	0.967
Tetrachloroethene	0.013	1.0	0.923
Toluene	0.014	1.0	0.931
1,1,1-Trichloroethane	0.023	1.0	0.967
1,1,2-Trichloroethane	0.036	1.0	0.937
Trichloroethene	0.012	1.0	0.903
Total Xylenes	0.149	3.0	1.01
Surrogate Compounds for GC/MS Method			
Toluene-d8	0.024	1.0	0.995
Bromofluorobenzene	0.090	1.0	1.11

D. INTERFERENCES

Tenax® is used as the sorbent in this method and is subject to artifact formation through reaction with compounds present in air such as nitrogen oxides (NO_x), ozone (O_3), and sulfur oxides (SO_x). Pellizzari et al. (1984) observed formation of benzaldehyde, acetophenone, and phenol at several micrograms per cartridge on exposure of virgin Tenax to parts per billion levels of O_3, nitric oxide (NO), nitrogen dioxide (NO_2), and chlorine. Trace levels of *a*-hydroxyacetophenone and ethylene oxide were also observed. These contaminants are also prone to increase when sampling ambient air with a high relative humidity. The presence of oxidants in the air can also produce artifacts by reaction with adsorbed organics. Walling et al. (1986) observed decomposition of pentachloroethane to tetrachloroethene during thermal desorption from Tenax.

Several compounds are commonly observed in blank, unexposed Tenax cartridges. Compounds observed by Environmental Science and Engineering, Inc. (ESE) and Walling et al. (1986) include benzene, toluene, ethylbenzene, methylene chloride, and benzaldehyde at levels of 5 to 30 nanograms (ng) per cartridge.

Solvents, reagents, glassware, and other sample-processing hardware may yield discrete artifacts and/or elevated baselines causing misinterpretation of chromatograms. These materials must be demonstrated to be free from interferences under the conditions of the analysis by running method blanks with each analysis lot.

E. ANALYSIS RATE

After instrument calibration, one analyst can analyze seven (7) Tenax or Tenax/carbon traps during a standard 8 hour work day.

F. SAFETY INFORMATION

These target compounds represent a wide range of chemical toxicity and suspected carcinogens. Only laboratory personnel with adequate experience in handling hazardous chemical constituents should be allowed to work with this procedure.

III. APPARATUS AND CHEMICALS

A. GLASSWARE/HARDWARE

1. Standard Preparation:
   a. 10-mL Class A volumetric flasks
   b. 2-dram vials with Teflon-lined screw caps
   c. 1-mL reactivials with stop-go caps
   d. Hamilton gas-tight syringes: 10, 50, 100, 250, 500, and 1,000 ul
   e. Microspatula
   f. Disposable pipets
   g. Analytical balance capable of weighing to 0.1 mg
   h. Soxhlet apparatus

GLASSWARE CLEANING: All glassware is to be detergent washed, and rinsed with tap water, DI water, acetone, and then hexane, followed by oven-drying at approximately 150°C for 1-2 hours. DI water is laboratory water that meets the requirements for ASTM Type I water.

2. Tenax® and Tenax®-charcoal Traps:

   a. 2,6-Diphenylene oxide polymer (Tenax®, 40/60 mesh).

   b. Granular Activated Charcoal (60/80 mesh), SKC petroleum-base or equivalent.

   c. Tenax Preparation: Prior to use, the 40/60 mesh Tenax resin is subjected to a series of solvent extraction and thermal treatment steps. All glassware used in Tenax purification as well as cartridge materials should be thoroughly cleaned by water rinsing followed by an acetone and methylene chloride rinse and dried in an oven at 180°C.

      Bulk Tenax is placed in a glass extraction thimble and held in place with a plug of clean glasswool. The resin is then placed in a soxhlet extraction apparatus and extracted sequentially with methanol and then pentane for 16-24 hours (each solvent) at approximately 6 cycles/hour. Glasswool for cartridge preparation should be cleaned in the same manner as the Tenax®.
      The extracted Tenax is immediately placed in an open glass dish and heated in an oven at approximately 40°C for two hours in a well ventilated area (a hood or a ventilated oven). The oven temperature is then increased to 110°C, using an inert gas flow, and held for one hour. The oven temperature control is then shut off and the oven is allowed to cool to room temperature. The Tenax that is not to be used immediately for cartridge preparation is stored in a clean glass jar having a Teflon-lined screw cap and placed in a desiccator.

   d. Cartridge Construction: The dimensions of the Tenax Cartridge and the typical sampling set-up is shown in Figure III.A.2.d-1 and Figure III A.2.d-2. This design uses a stainless steel construction, and eliminates the need to avoid direct contact with the exterior surface since only the interior of the cartridge is purged.

      All trap materials are pre-cleaned as described in Section III.A.2.c. The cartridge is packed by placing a 0.5-1 cm glasswool plug in the base of the cartridge and then filling the cartridge with 4 grams of Tenax. A 0.5-1 cm glasswool plug is placed in the top of the cartridge. Back-up traps are constructed by packing 2 grams of Tenax and then 2 grams of activated carbon into a stainless steel cartridge.

      Both front and backup cartridges are initially thermally conditioned by heating for twelve hours at 260°C under a nitrogen purge (50 ml/min). After the twelve hour heating period the cartridges are allowed to cool. Cartridges of the type shown in Figure III.A.2.d-1 are allowed to cool to room temperature

Figure III.A.2.d-1 and III.A.2.d-2

under inert gas purge and are then closed with brass plugs. Reconditioning of previously used traps involves conditioning for four hours at 260°C with a nitrogen purge flow of 50ml/min.

e. <u>Cartridge Preparation Quality Assurance</u>
Each batch of Tenax cartridges prepared is checked for contamination by analyzing one cartridge immediately after preparation. Analysis is accomplished by GC/MS and, for most compounds, the preparation blank level should be less than 5 nanograms per tube. If a cartridge does not meet these acceptance criteria, the entire lot is reconditioned. There are some common laboratory solvents (acetone and toluene) that are very difficult to remove from the tenax traps and for these compounds the blank concentrations should be less than 25 ng per tube or traps must be reconditioned.

f. Cartridges are to be used within 2 weeks of preparation and analyzed within two weeks of the sampling date. Cartridges are stored at -20°C in a clean freezer (i.e. no solvent extracts or other sources of volatile organics contained in the freezer).

B. INSTRUMENTATION

1. GC/MS: Hewlett Packard Model HP-5995C or equivalent; electron energy is 70 eV; mass range scanned is 35 to 350 m/e; analyzer temperature is 220°C.
2. Surrogate / Internal Standard / Calibration Standard Trap Spiking Accessory (TSA) - Figure III.B.2-1
3. Thermal desorption apparatus consisting of an aluminum heating block, a Nutech 320 controller, a Tekmar Model ALS automatic laboratory sampler, and a Tekmar Model LSC-2 liquid sample concentrator - Figure III.B.3-1, equipped with a 3-phase adsorbent trap (Silica gel/Charcoal/Tenax), Tekmar Co. part #14-4015-003.
   Run Conditions:
   Gas Flow Rate: 30 ml/min.
   Purge: 13 min at 29 °C or less.
   Desorb: 4 min at 180 °C.
   Bake: 25 min at 226 °C.

4. GC column: 30-meter by 0.53-mm ID DB-624 (3.0 micron film thickness) fused-silica megabore column

Figure III.B.2-1 and III.B.3-1

5. Oven Program:
   a. Hold isothermal at 20°C for 3 min
   b. Program at 4°C/min to 100°C
   c. Program at 20°C/min to 220°C and hold for 0.5min. Total run time is 29.5 minutes.
6. GC Conditions: Injection port: 220°C
   Transfer line: 240°C
   Carrier: Helium
   Flow rate: 8-10 mL/min (@ 100°C)
7. Retention times. A retention time window of ±0.3 min will be used for analytical purposes. Absolute and relative retention times for the analytes are listed in Section XII.

C. ANALYTES

Analyte	CAS Registry Number	Formula	Molecular Weight	Melting Point (°C)	Boiling Point(°C)
Benzene	71-43-2	C_6H_6	78	5	80
BCHPD	121-46-0	C_7H_8	92	*	89
Carbon tetrachloride	56-23-5	CCl_4	152	-23	77
Chlorobenzene	108-90-7	C_3H_5Cl	112	-45	132
Chloroform	67-66-3	$CHCl_3$	118	-63	61
1,1-Dichloroethane	75-35-4	$C_2H_4Cl_2$	98	-97	57
1,2-Dichloroethane	107-06-2	$C_2H_4Cl_2$	98	-35	83
trans-1,2-Dichloroethene	156-60-5	$C_2H_2Cl_2$	96	-50	48
DCPD	77-73-6	$C_{10}H_{12}$	132	-1	170
DMDS	624-92-0	$(CH_3)_2S_2$	94	-85	109
Ethylbenzene	100-41-4	C_8H_{10}	106	-95	136
MIBK	108-10-1	$C_6H_{12}O$	100	-80	117
Tetrachloroethene	127-18-4	C_2Cl_4	164	-22	121
Toluene	108-88-3	C_7H_8	92	-93	111
1,1,1-Trichloroethane	71-55-6	$C_2H_3Cl_3$	132	-35	75
1,1,2-Trichloroethane	79-00-5	$C_2H_3Cl_3$	132	-37	113
Trichloroethene	79-01-6	C_2HCl_3	130	-87	87
m-Xylene	103-38-3	C_8H_{10}	106	-47	139
o-Xylene	95-14-6	C_8H_{10}	106	-25	144
p-Xylene	25493-13-4	C_8H_{10}	106	-13	138

* Not available.

D. REAGENTS AND STANDARD ANALYTICAL REFERENCE MATERIALS (SARMS)

1. Methanol -- "Purge and Trap" quality or equivalent (Burdick and Jackson).

2. Spiking Solutions:

   a. The internal standards are Purgeables Internal Standards Mix (4-8835M) at 1000 ug/mL (Supelco - Bellefonte, PA). A secondary solution is prepared by adding 0.160 mL of the mix to a 4 mL volumetric and diluting to volume with methanol (final conc. = 40 ug/mL).

   b. The materials used for the surrogates are Volatiles Surrogate Spike Mix (4-8876M) at 250 ug/mL (Supelco). A secondary spiking solution is prepared by diluting 0.200 mL of the surrogate mix to 1 mL with methanol (final conc. = 50 ug/mL).

   c. The bromofluorobenzene (BFB) tune colution is prepared as follows: Into a tared 10 mL volumetric flask containing approx. 9 mL of reagent grade methanol inject 5 ul of neat 1-bromo-4-fluorobenzene (Aldrich, lot#01419CM, density=1.5269 g/mL). The nominal weight of this amount of surrogate is 0.0074g. The flask is then diluted to the mark with methanol, which generates a primary solution with a concentration of 740 ug/ml. From this primary solution a secondary solution is prepared by taking 170 uL of the primary and placing this aliquot into a 5.0 mL volumetric flask that is half filled with methanol. After diluting to the mark with methanol, this solution has a BFB concentration of 25 ug/mL.

   The parent mixes are accompanied by documentation that traces the identity and purity of the surrogate compounds to E.P.A. reference standards. For this reason, characterization of these mixes is not required.

3. All target analytes (except BCHPD) are USATHAMA supplied SARM compounds. BCHPD (bicyclo[2.2.1]hepta-2,5-diene) is obtained from Aldrich Chemical, Cat.# B3,380-3.

IV. CALIBRATION

A. INITIAL CALIBRATION

1. <u>Preparation of Standards</u>
   Stock primary calibration standards (PTS1-8) of the target compounds were prepared by measuring approximately 16 mg of each pure compound separately into a 10 mL volumetric flask and diluting to volume with methanol. This is accomplished (for the liquids) by adding calculated volumes of each analyte and determining the weight delivered based upon the density of the analyte (Table IV.A.1.1). These stock

calibration standards had concentrations of approximately 16,000 ug/mL for each analyte. A Primary Composite Tenax Calibration Standard (PCTCS) was prepared by adding approximately 0.625 mL of each primary solution to a 10 mL volumetric and diluting with methanol such that the final concentration of each analyte in the primary composite solution was 1000 ug/mL. Table IV.A.1.2 presents the working calibration standards for the target analytes. These standards should be prepared monthly and stored at 4°C.

Surrogate spiking solutions for the initial calibrations were made from the stock Supelco mix (Section III.D.2.b) as outlined in Table IV.A.1.3. These solutions are spiked concurrently with the analytical standards discussed above.

2. MS Intensity

Prior to GC/MS analysis, the MS will be tuned to meet the tuning criteria specified for bromofluorobenzene (BFB) according to the 2-88 Revision of the USEPA Contract Laboratory Program Statement of Work for Organics Analysis:

Mass	Required Intensity
50	15.0 - 40.0 percent of the base peak
75	30.0 - 60.0 percent of the base peak
95	base peak, 100 percent relative abundance
96	5.0 - 9.0 percent of the base peak
173	less than 2.0 percent of mass 174
174	greater than 50.0 percent of the base peak
175	5.0 - 9.0 percent of mass 174
176	greater than 95.0 percent but less than 101.0 percent of mass 174
177	5.0 - 9.0 percent of mass 176

Inject 50 ng BFB and if it meets the tune specifications proceed with analysis. Otherwise, reinject BFB and/or tune the mass spectrometer until the criteria are met. If necessary, mass calibrate the instrument with perfluorotertbutylamine (PFTBA) using a minimum of three ions, 69, 219, and 502. BFB tune must pass before any further analysis are conducted. No analysis of samples or standards can be completed until the MS tuning criteria has been met.

Table IV.A.1.1 Preparation of Primary Calibration Standards

Calibration Standard Prepared	Analyte to be Added	Volume of Standard Added (uL)	Density of Analyte	Weight of Analyte (mg)
PTS1	Ethyl Benzene	185	0.867	160
	Benzene	183	0.874	160
	Toluene	185	0.867	160
PTS2	MIBK	200	0.800	160
	DMDS	153	1.046	160
	Chlorobenzene	145	1.107	160
PTS3	1,1-Dichloroethane	136	1.177	160
	1,2-Dichloroethane	127	1.256	160
	trans-1,2-Dichloroethene	127	1.257	160
PTS4	1,1,1-Trichloroethane	120	1.338	161
	1,1,2-Trichloroethane	111	1.435	159
	Trichloroethene	110	1.462	161
PTS5	Methylene Chloride **	121	1.325	160
	Chloroform	107	1.492	160
	Carbon Tetrachloride	100	1.594	159
PTS6	DCPD	160 mg *		160
	Tetrachloroethylene	100	1.623	162
	BCHPD	190	0.854	162
PTS7	*o*-Xylene	180	0.897	161
	p-Xylene	185	0.866	160
	m-Xylene	185	0.868	161
PTS8	DBCP **	77	2.09	161

* DCPD is a solid at standard temperature and pressure
** Although methylene chloride and DBCP are not certified analytes, their includion into the standards facilitates the ability of the laboratory to report them as screened analytes.

Table IV.A.1.2. Preparation of Working GC/MS Calibration Standards

Calibration Standard Prepared	Standard to be Diluted	Volume of Standard (uL)	Final Volume (mL)	Final Concentration (ug/mL)
TSTD200	PCTCS	2000	10	200
TSTD050	PCTCS	500	10	50
TSTD010	PCTCS	100	10	10
TSTD002	PCTCS	20	10	2

Table IV.A.1.3. Preparation of Working GC/MS Surrogate Calibration Standards

Calibration Standard Prepared	Standard to be Diluted	Volume of Standard (mL)	Methanol Volume (mL)	Final Concentration (ug/mL)
TSUR200	#4-8876M	1.80	0.450	200
TSUR050	TSUR200	0.250	0.750	50
TSUR010	TSUR200	0.100	1.90	10
TSUR002	TSUR010	0.400	1.60	2

3. Instrument Calibration

Initial standard and surrogate calibration standards are prepared by injecting set volumes of the Working GC/MS Calibration and Surrogate Calibration Standards (Tables IV.A.1.2 and IV.A.1.3) into blank Tenax tubes (as prepared in Section III.2.d.) using the Trap Spiking Accessory (TSA) presented in Figure III.B.2-1. Amounts of each standard are found in Table IV.A.3.1. Precertification calibration is performed in duplicate utilizing the same standards and surrogates as in the initial calibration.

Standards are desorbed onto the GC/MS according to the following procedure:

a. A Tenax trap is connected to the TSA with 40 mL/min helium flow. 5 uL (200 ng) of internal standard mix (40 ug/mL, Section III.D.2.a) is injected onto the trap. The appropriate amount of standard/surrogate (Table IV.A.3.1) are injected onto the trap and flow is continued through the trap for an additional 2 minutes after spiking is completed.

b. The trap is removed from the TSA, the direction of flow reversed, and the trap attached to the Tekmar (Figure III.B.3-1). Flow through the column is maintained at 30 mL/min and the tube placed in the heated desorption block (@ 225°C) for 11 minutes. Analytes desorbed from the trap pass through 5 mL of organic free water in the sparger before being trapped on the internal Tekmar 3-phase trap (held at <30°C). This acts as a coolant and minimizes the problems associated with high temperature water being introduced onto the GC column. After every analysis the water in the sparge tube will be removed and the sparger will be rinsed once before another 5 mL of organic-free water is added to the sparger.

c. After the Tenax desorption cycle, the Tekmar trap is desorbed (185°C/4min) directly onto the DB-624 megabore and analysis completed per conditions outlined in Section III.B. NOTE: During the Tekmar desorption, the Tenax trap is removed from the heating block and allowed to cool to room temperature with helium flow. This prevents oxidative degradation of the Tenax allowing for future re-use.

4. Analysis of Calibration Data

Relative Response Factors (RRF) for all analytes at each amount (ng/tube) will be calculated using the equation below:

$$RRF = \frac{\text{Area of target analyte}}{\text{Area of internal standard}} \times \frac{\text{ng of internal standard}}{\text{ng of target analyte}}$$

Note that Table II.A.1 specifies which internal standards will be used for the normalization of all target analytes.

For each analyte, a percent relative standard deviation (%RSD) will be calculated from the RRF's injected for each certified reporting range. Two-thirds of the total analytes must meet a %RSD of less than 30% for the initial calibration to be considered valid. If more than one-third of the compounds exceed the 30% criteria, a new initial calibration must be analyzed.

The four Calibration Check Compounds (CCC's) listed below must also meet the 30% criteria for %RSD. If any of these compounds exceed this the 30%RSD limit, the initial calibration is considered invalid and must be repeated.

CCC's: 1,1-Dichloroethane
1,1,1-Trichloroethane
DMDS
o-Xylene

Table IV.A.3.1. Preparation of Initial Tenax Calibration Curve

Calibration Standard Prepared	Standard or Surrogate to be Used	Volume of Standard (uL)	Final Concentration (ng/tube)
TCC010	TSTD002/TSUR002	5	10
TCC020	TSTD002/TSUR002	10	20
TCC050	TSTD010/TSUR010	5	50
TCC100	TSTD010/TSUR010	10	100
TCC200	TSTD050/TSUR050	4	200
TCC500	TSTD050/TSUR050	10	500
TCC800	TSTD200/TSUR200	4	800
TCC1100	TSTD200/TSUR200	5.5	1100

B. DAILY CALIBRATION

1. Preparation of Standards
   A single calibration standard tube is prepared at 200 ng/tube per TCC200 presented in Table IV.A.3.1 except that 4 uL of surrogate spiking mix (50ug/mL, Section III.D.2.b) is injected onto the trap rather than the TSUR200.

2. MS Intensity
   Prior to the daily calibration, BFB is injected per Section IV.A.2, and the required intensities checked for adherence. Daily calibration and analysis of samples cannot proceed until the tuning criteria is met.

3. Instrument Calibration
   Injection of the daily calibration is completed per conditions outlined in Section IV.A.3.a-c. For the daily calibration and the daily method blank, two freshly conditioned Tenax traps will be used. The use of freshly conditioned traps minimizes the chance of residual amounts of target analytes in the tubes interfering with the one-point calibration or the method blank determination.

4. Analysis of Calibration Data
   Relative Response Factors (RRF) for all analytes will be calculated using the equation below:

$$RRF_D = \frac{\text{Area of target analyte}}{\text{Area of internal standard}} \times \frac{\text{ng of internal standard (200)}}{\text{ng of target analyte (200)}}$$

Note that Table II.A.1 specifies which internal standards will be used for the normalization of all target analytes.

For each analyte, the RRF_D will be compared to the average RRF_I calculated from the initial calibration. Two-thirds of the total analytes must meet a relative percent difference (%RPD), as calculated below, of less than 25% of the initial calibration for the daily calibration to be considered valid. If more than one-third of the compounds exceed the 25% criteria, a new daily calibration must be analyzed.

$$\%RPD = \frac{\overline{RRF_I} - RRF_D}{\overline{RRF_I}} \times 100$$

In addition, the four Calibration Check Compounds (CCC's) listed in Section IV.A.4 must meet a 25% criteria for %RSD. If any of these compounds exceed this limit, the daily calibration is considered invalid and must be repeated.

Should the second injection of the daily calibration not meet acceptance criteria, a new initial calibration must be performed.

V. CERTIFICATION TESTING

A. The primary spiking solutions for certification were prepared exactly as in Tables IV.A.1.1 and IV.A.1.2. Amounts to be spiked onto the Tenax tubes are presented in Table V.A.1. Analysis was completed as outlined in Section IV.A.3, and performed in duplicate on separate days. The analysis results of the certification spikes were processed by USATHAMA's IRPQAP certification program. The results are summarized in Attachment C.

Table V.A.1. Preparation of Certification Spikes

Certification Spike Prepared	Standard or Surrogate to be Used	Volume of Each Standard (uL)	Final Concentration (ng/tube)
TSP012	TSTD002/TSUR002	6	12
TSP030	TSTD010/TSUR010	3	30
TSP100	TSTD010/TSUR010	10	100
TSP300	TSTD050/TSUR050	4	300
TSP500	TSTD050/TSUR050	10	500
TSP600	TSTD200/TSUR200	3	600
TSP800	TSTD200/TSUR200	4	800
TSP1000	TSTD200/TSUR200	5	1000

VI. SAMPLING, HANDLING, AND STORAGE

A. SAMPLING PROCEDURE

Prior to sampling, the Tenax cartridges will be prepared according to instructions presented in Section III.A.2. In the field, sample traps of tenax and tenax/carbon are connected in series and then connected to the air sampling pump. The proper direction of air flow during sampling is indicated on both traps by an arrow that points in the direction of flow. Samples are collected at various flow rates and for various times depending upon the sensitivity desired, analyte breakthrough volumes and the concentrations of the analytes of interest. As an example, a flow rate of 100 ml/min for a time period of 8 h will usually provide sufficient sample to detect and quantitate concentrations of volatile analytes that are present in "typical" rural ambient air using a GC/MS in the scanning mode.

For ambient air measurements, samples should be collected in conjunction with meteorological data. In addition, the physical location of the traps, in relationship to the source, is extremely important to the interpretation to the tenax data.

Once samples are collected, the traps are disconnected from the sampling pump, and each other, and are recapped for transport to the laboratory (It is important to firmly cap all traps to avoid contamination during sample transport). Samples are packaged in a cooler and in blue ice during shipment to the laboratory. For every sampling period, one field blank (both the Tenax, and the Tenax/charcoal backup tibe) should be prepared and shipped to the laboratory with other field samples. If the field blank is found to contain traget analyte values that are >25% of the values found in the associated field samples, the laboratory will notify the contractor in charge of field sampling/data interpretation that significant field contamination has been discovered for this analysis.

B. CONTAINERS

Samples should be returned in coolers containing blue-ice. All end fittings on the tubes must be tightly fastened.

C. STORAGE CONDITIONS

All samples are to be stored in locked refrigerators at -4°C.

D. HOLDING TIME LIMITS

Holding times for analyses are 14 days from date of collection. If the sample tubes are not used within 14 days from conditioning, the tubes cannot be used for collection, but must be returned to the laboratory for reconditioning.

E. SOLUTION VERIFICATION

Verification of the calibration standards is based on the comparison of separately prepared sets of standards, made from separate primaries. Curves should agree within 10% in order to consider the curve preparation valid.

VII. PROCEDURE

A. EXTRACTION AND CLEANUP

The preparation of the Tenax and Tenax/carbon traps has been described previously.

B. CHEMICAL REACTIONS

Compounds containing bromine (i.e. DBCP) are prone to decomposition on hot metal surfaces.

C. INSTRUMENTAL ANALYSIS

1. Set up the analytical desorption apparatus shown in Figure III.B.3-1 and the TSA shown in Figure III.B.2-1.

2. Perform the instrument tune verification (BFB) and daily standard calibration outlined in Section IV.B. The tune verification and calibration are valid for 12 hours of analysis, from the time of BFB injection to the injection of the final sample.

3. Analyze a method blank (blank Tenax tube) according to the following protocols:

   a. The Tenax trap is connected to the TSA with 40 mL/min helium flow in the direction of the sampling was performed. 5 uL of internal standard mix (40 ug/mL, Section III.D.2.a) and 4 uL of surrogate spiking mix (50ug/mL, Section III.D.2.b) are injected onto the trap and flow is continued through the trap for an additional 2 minutes after spiking is completed.

   b. The trap is removed from the TSA, the direction of flow reversed, and the trap attached to the Tekmar (Figure III.B.3-1). Flow through the column is maintained at 30 mL/min and the tube placed in the heated desorption block (@ 225°C) for 11 minutes. Analytes desorbed from the trap pass through 5 mL of organic free water in the sparger before being trapped on the internal Tekmar 3-phase trap (held at <30°C). This acts as a coolant and minimizes the problems associated with high temperature water being introduced onto the GC column.

c. After the 11 minute Tenax desorption cycle, the Tekmar trap is desorbed (185°C/4min) directly onto the DB-624 megabore and analysis completed per conditions outlined in Section III.B. NOTE: During the Tekmar desorption, the Tenax trap is removed from the heating block and allowed to cool to room temperature with helium flow. This prevents oxidative degradation of the Tenax allowing for future re-use.

d. The results of the method blank are reviewed for indications of trap/system contamination. If no target analytes are detected above the certified reporting limits for each analyte, the method blank is considered valid and sample analysis can proceed.

4. Process samples according to the protocols outlined in Section VII.C.3 above.

5. Qualitative indentification is assigned by retention time and relative abundance of characterisitc ions for each target compound. Two criteria must be satisfied to verify the identifications: (a) elution of the sample component at the same GC relative retention time as the standard component, and (b) correspondence of the sample component and standard component mass spectra.

   a. For establishing correspondence of the GC relative retention time (RRT), the sample component RRT must compare within +/- 0.06 RRT units of the RRT of the standard component.

   b. Comparison of standard and sample component mass spectra are obtained and used for identification purposes, <u>only</u> if the GC/MS meets the daily tuning requirements for BFB. These standard spectra may be obtained from the run used to obtain reference RRTs. All ions present in the standard mass spectra at a relative intensity greater than 10% (most abundant ion in the spectrum equals 100%) <u>must</u> be present in the sample spectrum. The relative intensities of ions must agree within plus or minus 20% between the standard and sample spectra. (Example: For an ion with an abundance of 50% in the standard spectra, the corresponding sample abundance must be between 30 and 70 percent). Ions greater than 10% in the <u>sample</u> spectrum but not present in the <u>standard</u> spectrum must be considered and accounted for by the analyst making the comparison. The verification process should favor false negatives. All compounds meeting the identification criteria must be reported with their spectra. If a compound cannot be verified by all of the above criteria, but in the technical judgement of the mass spectral interpretation specialist, the identification is correct, then the compound is reported and compound quantification can then occur.

D. NON-CERTIFIED COMPOUND SEARCH

Non-certified compounds can be searched for using the Wiley-EPA Library and reported to 1 significant figure. Quantitation of non-certified analytes will be performed using the equation below:

$$\textit{Amount (ug)} = \frac{\textit{Response of unknown}}{\textit{Response of Internal Std.}} \times \textit{Concentration of Internal Std.}$$

These "hits" will be flagged with a "S" code before entry into the IRDMS Data base. A single internal standard (1,4-difluorobenzene) will be used for all calculations involving non-certified analytes. In addition, for these compounds that are reported as unknowns (identified as "UNK" in the PMRMA database), a relative retention time value will be calculated using this internal standard as the base retention time. A retention time value will be reported using the following equation:

Reported RRT = True RRT * 100

This value will be reported to the PMRMA database as "UNKXXX", where XXX is the reported retention time as calculated above.

E. ANALYSIS OF BACKUP TRAPS

At a minimum, 10% (one out of every ten sets) of the backup cartridges will be analyzed. The backup cartridge (Tenax/charcoal) from the sample with the highest amounts of target analytes in its front trap (Tenax only) will be analyzed. If this particular trap contains greater than 15% of the amount of components of interest found in the front cartridges, an additional trap will be analyzed. Should this trap exceed the 15% criteria, project management will be notified of the potential for trap saturation and the subsequent need to reduce sampling volume.

VIII. CALCULATIONS

From the instrument quantitation report, response factors for the target analytes will be determined according to the following equation:

$$RF = \frac{A_x}{A_{is}} \times I_s$$

where... A_x = Area response of the target compound
A_{is} = Area response of the internal standard
I_s = Amount of internal standard added in *u*g

These RF's will be used against the daily calibration RRF_D to quantify the total *u*g/tube for each compound as follows:

Analyte Amount (*u*g/tube) = RF / RRF_D

Note: The concentration of total xylenes will be calculated by taking the area counts of all isomer peaks and summing them to generate a single area count for the three isomers. This is the instrument response value that is to used to quantitate this analyte.

IX. DAILY QUALITY CONTROL

A. CONTROL SAMPLES

The control sample for this analysis shall consist of a method blank (blank Tenax) that will be prepared as specified in Section VI.A above, spiked with the two surrogates. The standard sample will be processed and analyzed in exactly the same manner and at the same time as all the other samples in the analysis lot. This control sample is to be analyzed immediately after the daily calibration for each analysis lot.

For this analysis method, the surrogates listed in Table II.A.1 will be the control analytes. Each Tenax trap will be spiked with 200 ug of the individual surrogates.

B. CONTROL CHARTS

Control charts are used to monitor the variations in the precision and accuracy of routine analyses and detect trends in these variations. The construction of a control chart requires initial data to establish the mean and range of measurements. The QC control charts are constructed from data representing performance of the complete analytical method. Data used in control charts shall not be adjusted for accuracy.

In the initial construction of the control charts, data from the laboratory certification analyses will be used. Data from the spiked QC sample within an analytical lot will be compared to control chart limits to demonstrate that analyses of the lot are under control, and will be used to update the charts. Control charts are prepared for all of the six surrogate analytes using the percent recovery data calculated according to the following equation:

$$\text{Percent Recovery} = \frac{\text{Found Concentration}}{\text{Spiked Concentration}} \times 100 \text{ percent}$$

Note that during the analysis of the certification data, none of the target analytes were found in the unspiked Tenax trap. Therefore blank correction should not be necessary.

Preparation of control charts requires the following data:

1. Three-point moving average percent recovery (X) for each surrogate in the spiked QC sample in the analytical lot; and,

2. Three-point moving range for the percent recovery of each surrogate in the spike QC sample.

X. REFERENCES

1. "Determination of Volatile Organic Compounds in Ambient Air Using Tenax® Absorption and Gas Chromatography/Mass Spectrometry (GC/MS), Method TO-1, Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air, EPA/600/4-89/017, June 1988.

2. Pellizzari, E., Demian, B., and Krost, K. 1984. "Sampling of Organic Compounds in the Presence of Reactive Inorganic Gases with Tenax GC." Anal. Chem., 56(4), 793-798.

3. Walling, J.F., Bumgarner, J.E., Driscoll, D.J., Morris, C.M., Riley, A.E., and Wright, L.H. 1986. "Apparent Reaction Products Desorbed from Tenax Used to Sample Ambient Air." Atmospheric Environment, 20(1), 51-57.

XI. DATA

A. OFF-THE-SHELF ANALYTICAL REFERENCE MATERIALS CHARACTERIZATION

All target analyte standards materials used for this certification were SARM's supplied from the USATHAMA SARM repository except BCHPD which was obtained from Aldrich Chemical. The surrogate and internal standard spiking solutions are supplied from the manufacturer with documentation that traces them to E.P.A. reference standards. Because of this, these materials do not require characterization.

B. INITIAL CALIBRATION/PRECERTIFICATION DATA

Refer to Attachment A. for a presentation of the precertification data that was generated for this method.

C. DAILY CALIBRATION

Attachment B. contains the calibration data that was generated for the analysis of the certification spikes. Also included with the calibration data are the reference mass spectra (from the Wiley Library) and the standards mass spectra for each target compound, surrogate, and internal standard.

D. STANDARD CERTIFICATION SAMPLES

Attachment C. presents the certification data results generated by the USATHAMA IRPQAP program for this method.

Attachment D. contains chromatograms and quantitation reports that were generated during the analysis of the certification spikes. Contained in this attachment are two chromatograms with data for the highest tested concentration spikes and the two certification spikes at the 20 ng/tube levels.

XII. RETENTION TIME DATA

Compound	Retention time (min)	Relative Retention Time (to nearest I.S.)
Bromochloromethane*	7.63	1.00*
Methylene Chloride	4.66	0.610
trans-1,2-Dichloroethene	5.12	0.671
1,1-Dichloroethane	5.94	0.779
Chloroform	7.92	1.039
1,1,1-Trichloroethane	8.11	1.063
Carbon Tetrachloride	8.44	1.106
Benzene	8.96	1.175
1,2-Dichloroethane	9.13	1.196
1,4-Difluorobenzene*	10.15	1.00*
Bicycloheptadiene	10.61	1.045
Trichloroethene	10.65	1.050
DMDS	13.26	1.307
Toluene-d8(S)	13.85	1.365
MIBK	13.87	1.366
Toluene	14.02	1.381
1,1,2-Trichloroethene	15.36	1.514
Tetrachloroethene	15.49	1.526
Chlorobenzene-d5*	18.04	1.00*
Chlorobenzene	18.13	1.005
Ethylbenzene	18.57	1.029
m- and p-Xylene	18.97	1.052
o-Xylene	20.16	1.118
Bromofluorobenzene	21.84	1.211
Dicyclopentadiene	24.79	1.374
DBCP	26.41	1.464

* Internal Standard
(S) Surrogate