Cook
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HW CA

Enseco
A CORNING Company

QUALITY ASSURANCE PROJECT PLAN
for
COOK COMPOSITES AND POLYMERS CO.
(formerly Freeman Chemical Corporation)

Submitted: January 1991

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1. INTRODUCTION

This quality assurance program plan (QAPP) outlines specific quality assurance (QA) and quality control (QC) procedures to be followed by Enseco - Erco Laboratory (Erco) regarding chemical analyses related to Freeman Chemical Corporation's study and monitoring of groundwater at their manufacturing plant in Saukville, Wisconsin. The plan calls for the analysis of groundwaters for Appendix IX constituents and the analysis of water, soil, and sludge for volatile organic compounds (VOC) and semivolatile compounds (SVC).

2. DEFINITION, PURPOSE, AND SCOPE

2.1 Definition of Terms

The following terms are defined for the purpose of this QAPP and the study involved.

- Appendix IX Hazardous Constituents - Compounds listed in 40 CFR Part 264, which pertain to groundwater monitoring under the Resource Conservation and Recovery Act (RCRA).
- Quality Assurance - The total integrated program designed to ensure the reliability of data generated in the laboratory.
- Quality Control - The routine application of specific, well-documented procedures to ensure the generation of data that is of known and acceptable quality, which will fulfill the objectives of the QA program.
- Standard Operating Procedure (SOP) - A detailed, written description of a procedure designed to systematize and standardize the performance of the procedure.
- Quality Assurance Project Plan - An assemblage of detailed SOPs which describe how the laboratory will generate data that meet the data quality objective of a specific project.

2.2 Purpose

The purpose of this QAPP is to provide a detailed description of all elements involved in the generation of data of acceptable quality and completeness for the monitoring of VOCs and SVCs in water, soil, and sludge and Appendix IX constituents in groundwater. Guidelines for this plan have been obtained from "Interim Guidelines and Specifications for Preparing Quality Assurance Project Plans," Office of Monitoring Systems and Quality Assurance, Office of Research and Development, U.S. Environmental Protection Agency (U.S. EPA), EPA-600/4-83-004, February 1983.

2.3 Scope

The scope of this QAPP is to outline QC requirements for all data generated during the project based on quality judgements using the following three types of information.

- Overall qualifications data which includes internal and external performance and systems audits to ensure that there are adequate facilities and equipment, qualified personnel, documented laboratory procedures, accurate data reduction, proper validation, and complete reporting.
- Data that measure the daily performance of the laboratory according to the specific method employed. This includes data on calibration procedures and instrument performance.
- Data that evaluate the overall quality of the package that is used to determine precision, accuracy, representativeness, completeness, and comparability which is in compliance with the data quality objectives listed in section 12. Such data includes laboratory blanks, field blanks, laboratory control spikes, laboratory control spike duplicates, and surrogate spike recoveries.

3. PROJECT ORGANIZATION AND RESPONSIBILITIES

In order to ensure that all QA/QC procedures are strictly adhered to, specific responsibilities must be assigned to each individual involved in the project. The QA director, Dennis Flynn, will oversee and be responsible for all QA/QC activities including audits, preparation of QA specifications, and corrective actions. Mr. Flynn reports directly to Jack Farrell, the Erco division director, and to Peggy Sleevi, the Enseco QA director. Laboratory managers are responsible for producing fully documented data of acceptable quality from their respective laboratories. Figure 3-1 illustrates the Erco organizational structure.

Gas chromatography/mass spectrometry (GC/MS) tentatively identified compounds will be reviewed by Peter Kane and Daniel Wielandt, managers of the laboratories, or by senior personnel in the data review groups to whom they designate the task.

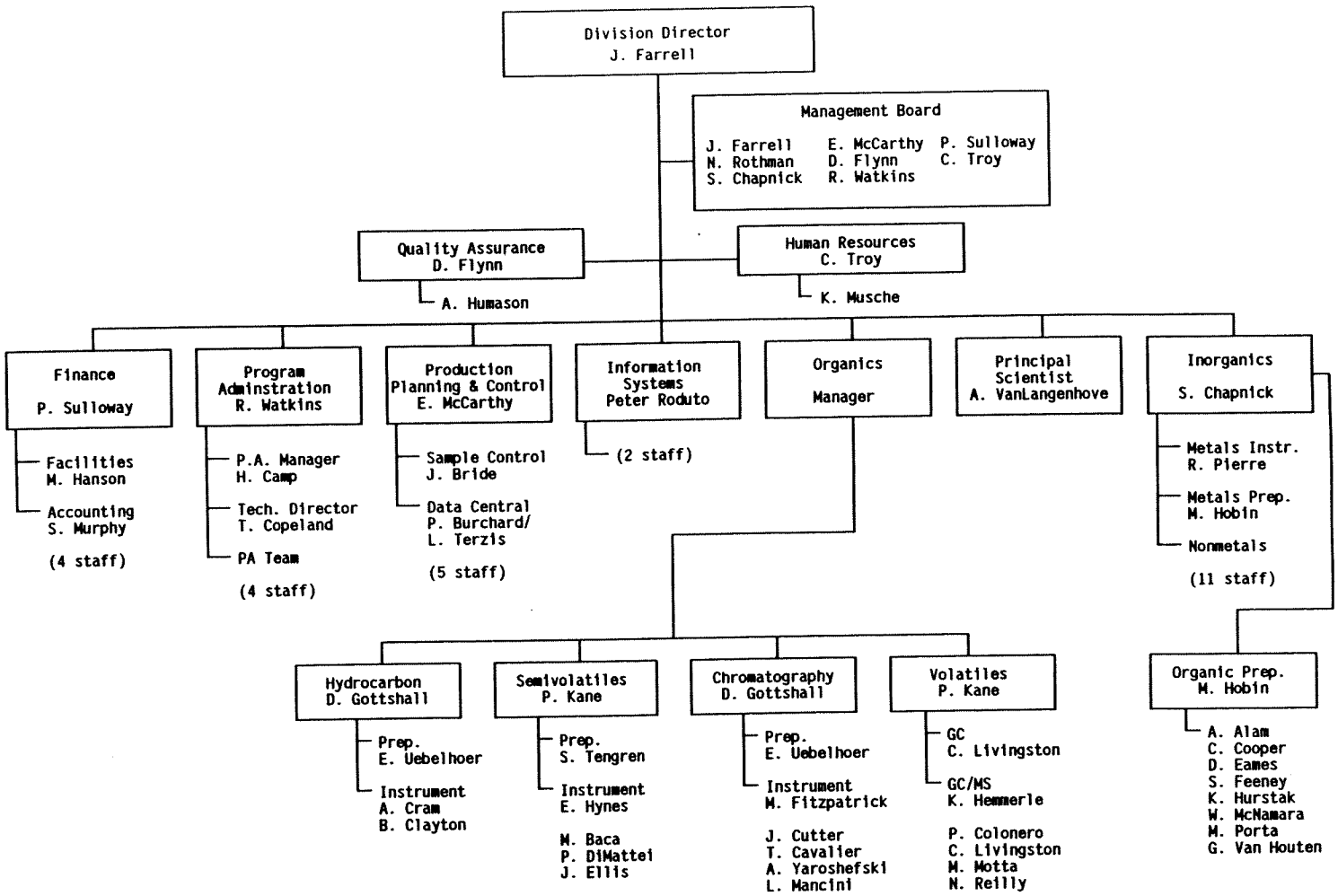


Figure 3-1. Erco Organizational Chart.

4. SAMPLING PROCEDURES

4.1 Sample Containers, Preservatives, and Holding Times

Sample containers, preservatives, and holding times for VOCs, SVCs, and additional Appendix IX constituents are outlined in Tables 4-1 and 4-2. Erco will provide appropriately prepared sample containers and coolers for this project. All containers are purchased precleaned and prebaked from I-Chem of Hayward, California. The containers are 200 series bottles that are cleaned for the particular analyses according to U.S. EPA specifications.

4.2 Sample Packing, Handling, and Shipping

After a sample is collected, a self-adhesive label will be prepared with indelible ink and affixed to each container. At a minimum, the sample label will contain the following items of information.

- Investigation name: Hatcher-Sayre/Freeman Chemical Corporation.
- Field sample number.
- Sample description.
- Date and time collected.
- Sampler's initials.
- Testing required.
- Preservative.

Immediately after sample collection, each labeled sample container will be sealed in an individual plastic bag. Samples will immediately be placed into an insulated cooler with ice or ice packs for shipment to the laboratory.

Chain-of-custody (COC) procedures are outlined in Section 5. COC records are sealed in zip-lock bags to protect them from moisture and will then be

Table 4-1. Recommended containers, preservatives and holding times for monitoring for VOCs and SVCs (base-neutral acid extractables and pesticides/polychlorinated biphenyls [PCB])*

Sample Container	Preservation	Minimum Sample Size	Parameters/Methods	Recommended Holding Times**
		Aqueous Samples		
3 x 40-mL glass (VOA)	4°C, HCl to pH <2	40 mL	Volatile Organics/ Method (5030) 8240	14 days
3 x 40-mL glass (VOA)	4°C, HCl to pH <2	40 mL	Aromatic Volatile Organics/ Method (5030) 8020	14 days
2 x 1-liter glass	4°C	1,000 mL	Semivolatile Organics/ Method (3510) 8270	7 days until extraction; 40 days after extraction
		Solid Samples		
3 x 40 mL glass (VOA)	4°C	20 g	Volatile Organics/ Method (5030) 8240	14 days
3 x 40 mL glass (VOA)	4°C	20 g	Aromatic Volatile Organics/ Method (5030) 8020	14 days
2 x 8-oz jar, glass	4°C	100 g	Base-neutral Acid Extractables/ Method (3540), (3550) 8270	7 days until extraction; 40 days after extraction

Please note that no filtration is required for VOCs or SVCs.

*Preparation methods are in parentheses, followed by analytical procedure (where applicable).

**Holding time is calculated from the date of sample collection.

Table 4-2. Recommended containers, preservatives and holding times for Appendix IX constituents*

Sample Container	Preservation	Minimum Sample Size (mL)	Parameters/Methods	Recommended Holding Times**
2 x 1-liter glass	4°C	1,000	Organochlorine, Organophosphorous, Pesticides/ Method (3510), (3520) 8080, Method (3510), (3520) 8140	7 days until extraction; 40 days after extraction
2 x 1-liter glass	4°C	1,000	Herbicides/ Method 8150	7 days until extraction; 40 days after extraction
2 x 1-liter glass	4°C	1,000	Dioxins, Furans/ Method 8280	7 days until extraction; 40 days after extraction
3 x 40-mL glass (VOA)	4°C, HCl to pH <2	40	Volatile Organics/ Method (5030) 8240	14 days
2 x 1-liter glass	4°C	1,000	Semivolatile Organics/ Method (3510), (3520) 8270	7 days until extraction; 40 days after extraction
1 x 0.5-liter polyethylene	HNO_3 to pH <2***	100	Total Metals, ICP/ Method (3010) 6010	6 months
1 x 0.5-liter polyethylene	HNO_3 to pH <2***	100	Total Metals, Furnace (As, Se, Tl, Pb)/ Methods (3020) 7060 7740, 7841, 7421	6 months

Please note that no other Appendix IX constituents require filtration.

*Preparation methods are in parentheses, followed by analytical procedure (where applicable).

**Holding time is calculated from the date of sample collection.

***This preservation is for total metals. Dissolved metals require filtration prior to the pH adjustment.

Table 4-2. Continued

Sample Container	Preservation	Minimum Sample Size (mL)	Parameters/Methods	Recommended Holding Times**
1 x 0.5-liter polyethylene	HNO_3 to pH <2***	100	Mercury, CVAA/ Method 7470	28 days
Polyethylene	NaOH to pH>9 4°C, Zn $(C_2H_3O_2)_2$	100	Total Sulfide/ Method 9030	7 days
Polyethylene	NaOH to pH >12, 4°C	250	Total Cyanide/ Method 9010	14 days

Please note that no other Appendix IX constituents require filtration.

**Holding time is calculated from the date of sample collection.

***This preservation is for total metals. Dissolved metals require filtration prior to the pH adjustment.

taped to the underside of the appropriate sample cooler door. Each cooler must contain sufficient ice and/or ice packs to ensure that the proper temperature is maintained. Each cooler will be packed with the adequate packing material (e.g., vermiculite) required to prevent damage to the sample containers.

The sample coolers will be shipped by an overnight courier according to current U.S. Department of Transportation (DOT) regulations.

5. SAMPLE CUSTODY

The sample chain of custody is initiated at the laboratory when sample containers are sent from the laboratory to the sampling site. Sample containers are sent in sealed coolers along with COC forms (figure 5-1). All shipments are recorded in the cooler request logbook (figure 5-2). Once the samples are received at the laboratory, the sampling personnel who receive the cooler sign the airbill which serves as the documentation. The sampling team then fills and labels each bottle (figure 5-3). The samples are properly packed in the cooler and the cooler is sealed (figure 5-4) and shipped.

Samples are received by the Erco sample custodian who records all incoming shipments in the incoming shipping log (figure 5-5) and the sample control worklist (figure 5-6). The coolers are then unpacked and all samples are inspected for breakage, proper preservation, and accurate and complete paperwork. The sample control project checklist is then completed (figure 5-7). Once the samples are checked in, they are logged into the laboratory information management system (LIMS) database. When log-in procedures are complete, LIMS generates a group codes screen print report (figure 5-8) and a sample list screen print report (figure 5-9). These screen print reports, together with the project checklist, are brought to the program administrator (PA) for inspection and approval.

Once everything is approved by the PA, the sample custodian generates a labtrack form (figure 5-10) from LIMS for each of the Erco laboratories involved with the particular project. Each laboratory signs the appropriate line on the labtrack and the sample custodian signs it as well. The laboratory also signs the internal chain-of-custody sample tracking log (figure 5-11) when the samples are picked up. All samples are kept in a secure, locked refrigerator in the sample control area until they are picked up by the laboratory.

Enseco-Erco Laboratory

205 Alewife Brook Parkway
Cambridge, Massachusetts 02138
617/661-3111 Fax: 617/354 5258

Attn: _____

CHAIN OF CUSTODY

No. 9064

SAMPLE SAFE™ CONDITIONS

1. Packed by: _____ Seal # _____
2. Seal Intact Upon Receipt by Sampling Co. Yes No
3. Condition of Contents: _____
4. Sealed for Shipping by: _____
5. Initial Contents Temp.: _____ °C Seal # _____
6. Sampling Status: Done Continuing Until _____
7. Seal Intact Upon Receipt by Laboratory: Yes No
8. Contents Temperature Upon Receipt by Lab: _____ °C
9. Condition of Contents: _____

Enseco Client _____

Project _____

Sampling Co. _____

Sampling Site _____

Team Leader _____

Date	Time	Sample ID/Description	Sample Type	No. Containers	Analysis Parameters	Remarks

CUSTODY TRANSFERS PRIOR TO SHIPPING

	Relinquished by: (signed)	Received by: (signed)	Date	Time
1				
2				
3				

SHIPPING DETAILS

Delivered to Shipper by: _____

Method of Shipment: _____ Airbill # _____

Received for Lab: _____ Signed: _____ Date/Time _____

Enseco Project No. _____

White and Pink Copies to Lab Yellow to Sampler

SS-001 (CM)

Figure 5-1. Chain-of-Custody Form.

COOLER REQUEST LOG BOOK

Date:_____

Note: All request must be logged into this book. Any request brought down after 1600 must be pre-approved. No exceptions will be made as this rule is for the benefit of the client.

Client Name	PA	Project #	Date needed to client	Time in to SC	Completed by Date/Initial	Cooler Number

Figure 5-2. Cooler Request Logbook.

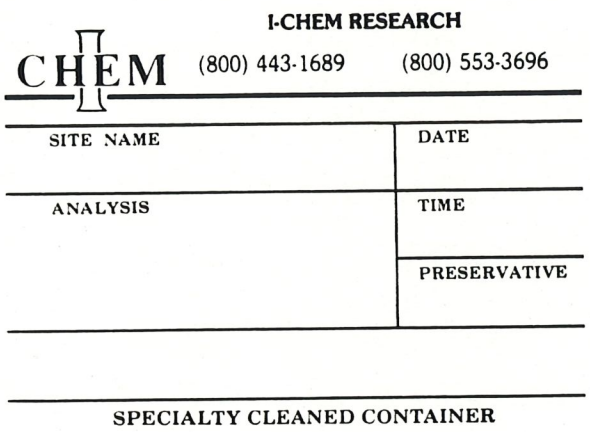
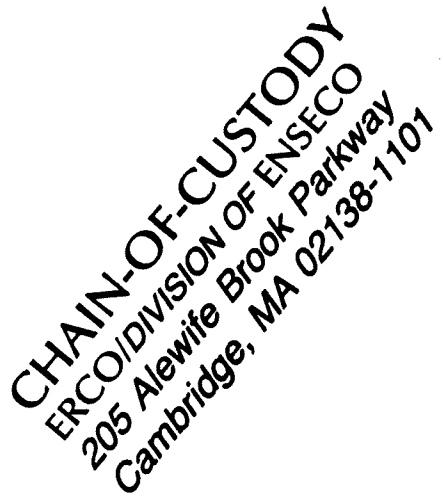


Figure 5-3. Sample Label.



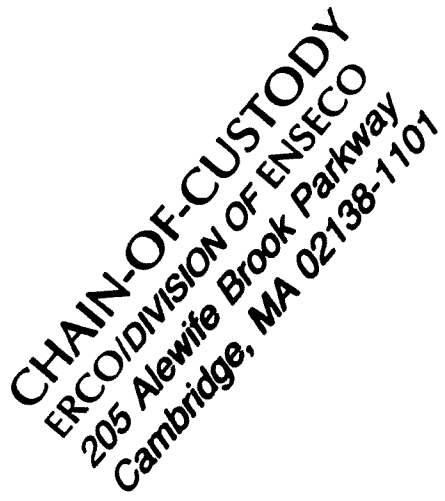


Figure 5-4. Cooler Seal.

Erco Shipping Log - Incoming

Page ___

Date	Time	Carrier	Airbill Number	Name of Company	Shipment for	Received by (signature)	Item	COD

Figure 5-5. Incoming Shipping Log.

Sample Control Worklist

Date:_____

It is the policy of the Sample Control Area to assure a twenty four(24) hour turnaround time for Sample Login. All projects received by morning couriers will be logged in, have paperwork generated and be distributed to the PAs by 12:00 (noon) each day. All projects received throughout the day will be logged in and distributed ASAP.

Project Number	PA	Delivery Method	Client	Analyses Required

Figure 5-6. Sample Control Worklist.

Enseco - Erco Sample Control Project Checklist

Erco Project Number: _____ Please check project type:

Date Received: _____ Enseco ___ Rush ___

Client: _____ CLP ___ Standard ___

Contact: _____ Tier I ___ Extended ___

PA: _____ Tier II ___

___ Cooler(s)/ID No. _____ Enseco ___ Erco ___ Client ___

Type of Bottles: Enseco ___ Erco ___ Client ___

Courier/Delivered by: _____

Airbill Present: Yes __ No __ Custody Seals Intact: Yes __ No __

Traffic Report Present: Yes __ No __ Chain of Custody Present: Yes __ No __

LIMS project initiated: Yes __ No __

Enter Project History information as Sample Delivery Acknowledgement:

Temperature of cooler upon receipt was...
Bottles were/not broken in transit.
Bottles were/not properly labeled (yes or no).
Samples agree with Chain-of-Custody (yes or no).
Samples were properly preserved (yes or no).
VOA Vials were properly preserved (yes or no).
VOA Vials did/did not contain headspace (yes or no).

Short Holding Times: Yes __ No __ Metals need filtering: Yes __ No __

Subcontract Parameters: Yes __ No __ Aliquoting/compositing: Yes __ No __

(Short holding times and subcontracting must be documented appropriately)

Chain of Custody Signed: Yes __ No __ Sample Picture Taken: Yes __ No __

SC Project Checklist Completed: _____ Date: _____

Bottle Labeling Completed: _____ Date: _____

PA review - Given the project information available, did the SC successfully log in the project upon the first try: Yes __ No __

Error: _____ Solution: _____

PA Sample Delivery Acceptance: _____ Date: _____

Figure 5-7. Sample Control Project Checklist.

ENSECO, Inc.
Date: 30 JAN 91

Erco Laboratory
Group Codes Screen Print Report
Page 1

GROUP CODE
Client: ERCO -HAI-VA00 Name: Hatcher-Sayre, Inc.
Program: ERCO -003274 Name: HAI-KY00 Freeman Chemical Monitoring 1991
Project: ERCO -007933 Name: HAI-KY00 Freeman Chemical Monitoring 01/10/91

Grp	Smp#	Description	Matrix	Disposal	Inst
A	15	624	AQUEOUS	PENDING	0
B	4	624,Phenols	AQUEOUS	PENDING	0
C	9	602	AQUEOUS	PENDING	0

Grp	Description	Matrix	Disposal	# Samples Expect
A	624	AQUEOUS	PENDING	15

Package/Project	Test Code	Vr	Rp	Test Description	Inst
	624-TCL-A	1	0	Method 624 - TCL Volatile	0 0

Package/Project	Test Code	Ver	Rp	Test Description
	624-TCL-A	1	0	Method 624 - TCL Volatile Org

Grp	Description	Matrix	Disposal	# Samples Expect
B	624,Phenols	AQUEOUS	PENDING	4

Package/Project	Test Code	Vr	Rp	Test Description	Inst
	624-TCL-A	1	0	Method 624 - TCL Volatile	0 0
	PHEN-A	1	0	Phenolics	0
	P-PHEN-A	1	0	Prep - Phenolics	0

Grp	Description	Matrix	Disposal	# Samples Expect
C	602	AQUEOUS	PENDING	9

Package/Project	Test Code	Vr	Rp	Test Description	Inst
	602-A	2	0	Method 602 - Aromatic Volat	0

Package/Project	Test Code	Ver	Rp	Test Description
	602-A	2	0	Method 602 - Aromatic Volatil

Figure 5-8. Group Codes Screen Print Report.

ENSECO, Inc.
Date: 30 JAN 91

Erco Laboratory
Sample List Screen Print Report
Page 1

PROJECT (Page 1 of 2)
Client: ERCO -HAI-VA00 Name: Hatcher-Sayre, Inc.
Program: ERCO -003274 Name: HAI-KY00 Freeman Chemical Monitoring 1991
Project: ERCO -007933 Name: HAI-KY00 Freeman Chemical Monitoring 01/10/91
Type: ENGINEER Case: SDG: SAS: Level:
PO #: Invoice #:
Description:

Contact: ERCO -HAI-VA06 Hatcher-Sayre, Inc. Money (804)794-0216
Alternate: - ( ) -
Rpt Cpy: - ( ) -
Billing: ERCO -HAI-VA01 Hatcher-Sayre, Inc. Payable (804)794-0216
E-Mail: - ( ) -
Project Status: INVOICED Status History: IMDASB Probability: 1.00
Case: SDG: SAS: IEC's: BG Corr's: BG Before:
Results to client computer? N Deliverables: COMM
Phone results to client? N Client Project ID:
Other ENSECO lab involved? N
Subcontractor involved? N Sales Exec:
Calculate holding times from collection dates? Y Primary PA: MFORD
Delivery Penalty: NONE Turnaround Class: NORMAL
Last Followup Date: 29 OCT 90 Promised Turnaround Days: 30
Actual Start Date: 10 JAN 91 Next Followup Date:
Project Due Date: 09 FEB 91 Expected Start Date: 10 JAN 91
Receipt Date: 10 JAN 91 Time: 12:00 Storage Location:
Comments:

Log Released: MFORD 11 JAN 91 Invoiced: SMURPHY 23 JAN 91
Cooler Shipped: Reported: LCULLEN 23 JAN 91
Delivery Accepted: AHERNANDEZ 10 JAN 91 13:25 Reviewed: LCULLEN 23 JAN 91
Created: MFORD 31 DEC 90 Modified: SMURPHY 24 JAN 91

Sm#	Grp	Alq	Rec Date	Due Date	Matrix	Status	Client Description	Inst
0001	C	SA	10 JAN 91	06 FEB 91	AQUEOUS	INVOICED	W-41	0
0002	A	SA	10 JAN 91	06 FEB 91	AQUEOUS	INVOICED	W-14B	0
0003	A	SA	10 JAN 91	06 FEB 91	AQUEOUS	INVOICED	W-6A	0
0004	C	SA	10 JAN 91	06 FEB 91	AQUEOUS	INVOICED	W-42	0
0005	C	SA	10 JAN 91	06 FEB 91	AQUEOUS	INVOICED	W-47	0
0006	A	SA	10 JAN 91	06 FEB 91	AQUEOUS	INVOICED	W-23	0
0007	C	SA	10 JAN 91	06 FEB 91	AQUEOUS	INVOICED	RC-1	0
0008	C	SA	10 JAN 91	06 FEB 91	AQUEOUS	INVOICED	RC-2	0
0009	C	SA	10 JAN 91	06 FEB 91	AQUEOUS	INVOICED	RC-3	0
0010	A	SA	10 JAN 91	06 FEB 91	AQUEOUS	INVOICED	W-30	0
0011	A	SA	10 JAN 91	06 FEB 91	AQUEOUS	INVOICED	W-30 DUP	0
0012	A	SA	10 JAN 91	06 FEB 91	AQUEOUS	INVOICED	FIELD BLANK 1/8/91	0
0013	A	SA	10 JAN 91	06 FEB 91	AQUEOUS	INVOICED	MW-1	0
0014	A	SA	10 JAN 91	06 FEB 91	AQUEOUS	INVOICED	MW-2	0
0015	A	SA	10 JAN 91	06 FEB 91	AQUEOUS	INVOICED	MW-3	0

Figure 5-9. Sample List Screen Print Report.

KAN1.TXT;1 May 15 11:35 1990 ,LDMS$D Page 1

1/87 Rev.

FORM I SV-2
ENSECO-ERCO LABORATORY

VOA-GCMS Lab Tracking/Anomaly Form

Type:

Page: 1

Booking # : 000000 Date Due : APR 14 1990
PA : DFLYNN

Client : Enseco, Incorporated
Date Rec'd: 28 FEB 90

ERCO ID & Aliquot	Client ID	Matrix	Test(s)	Coll. Date	Prep Date	Date Run	Instr.	Associated Blank	FRN#	Anomalies
005672-0001-SA	Sample # 1	AQUEOUS	624-TCL-A	FEB 28 1990						
005672-0002-SA	Sample # 2	AQUEOUS	624-TCL-A	FEB 28 1990						
005672-0003-SA	Sample # 3	AQUEOUS	624-TCL-A	FEB 28 1990						

Project Description: VOA'S: 624'S not preserved.
voa-o 3/6/90 ms

Associated DCS(s)

Comments:

Samples Rec'd By Lab_____ Date_____

Samples Transferred to Instrument Lab_____ Date_____

Initial Review_____ Date_____

Final Technical Review_____ Date_____

Data Entered_____ Date_____

Data Transcription Review_____ Date_____

ERCO Auditor_____ Date_____

Enseco Auditor_____ Date_____

Figure 5-10. Labtrack Form.

Enseco - Erco Laboratory
Internal Chain of Custody
Sample Control Tracking Log

Project Number	Project Acceptance Date	***Laboratory Acceptance Boxes***						
		Metals	Nonmetals	Herb/ Pest	Hydro	BNA	VOA GC	VOA GC/MS
8563								
8564								
8565								
8566								
8567								
8568								
8569								
8570								
8571								
8572								
8573								
8574								
8575								
8576								
8577								
8578								
8579								
8580								
8581								
8582								
8583								
8584								

NOTE: Please sign name and date to confirm laboratory acceptance. Sample Control must highlight the Laboratory Acceptance Box to indicate which laboratories are affected.

Figure 5-11. Internal Chain-of-Custody Sample Tracking Log.

Once sample analysis is complete, all samples and extracts are stored for 90 days from the date of reporting. Once the 90-day period has expired, the samples, along with the containers, are disposed of by incineration at a licensed hazardous waste treatment, storage, and disposal facility. All tags and identification are removed from the samples before disposal and are placed in the permanent file for the project.

6. CALIBRATION PROCEDURES AND FREQUENCY

Erco will employ analytical methods found in "Test Methods for Evaluation of Solid Waste," (SW-846), Office of Solid Waste and Emergency Response, U.S. EPA; 3rd edition. Methods contained in this manual cite the specific calibration and check procedures that are required to conduct the analyses properly. Examples of these specific procedures follow in this section.

6.1 Gas Chromatography/Mass Spectrometry -- Volatiles

The instrument is hardware-tuned using 50 ng of 4-bromofluorobenzene (BFB). Ion abundance criteria must meet that of the EPA Contract Laboratory Program (CLP) protocols. Initial calibration is required at 20 µg/L, 50 µg/L, 100 µg/L, 150 µg/L, and 200 µg/L. Average response factors (RF) and relative standard deviations (RSD) are calculated for each compound (see table 7-1 for the target compound list [TCL]). Calibration check compounds (CCC) are 1,1-dichloroethane, chloroform, 1,2-dichloropropane, toluene, ethylbenzene, and vinyl chloride. System performance check compounds (SPCC) are chloromethane, 1,1-dichloroethane, bromoform, 1,1,2,2-tetrachloroethane, and chlorobenzene. For the initial calibration to be considered valid, the RSD must be less than or equal to 30.0% for CCCs. The RF for SPCCs must be 0.300 or greater (0.250 or greater for bromoform). Analysis of samples can proceed for 12 hours following the time of the BFB injection once these criteria are met. Continuing calibration is achieved through 50 ng BFB injections and criteria, and a calibration standard containing all of the TCL compounds. For a continuing calibration to be valid, SPCCs must meet the same criteria as that for the curve, and CCCs RFs less than or equal to 25.0% difference from those of the curve. Sample analysis can proceed for 12 hours from the time of the BFB injection once these criteria are met.

6.2 Gas Chromatography/Mass Spectrometry -- Semivolatiles

Semivolatile calibration procedures follow the same analytical calibration scheme as that of volatiles with the following differences. Decafluorotriphenylphosphine (DFTPP) is used to meet ion abundance criteria. The initial calibration is required at 20 ng/μL, 50 ng/μL, 80 ng/μL, 120 ng/μL, or 160 ng/μL. Nine compounds contained on the TCL are not required in the 20 ng/μL standard. There are 13 CCCs and 4 SPCCs that are listed in the method. The minimum acceptable RF for SPCCs is 0.050. The maximum percent RSD for the CCCs in the initial calibration is 30.0%. The percent difference required for continuing calibrations is 30.0%.

6.3 Standards

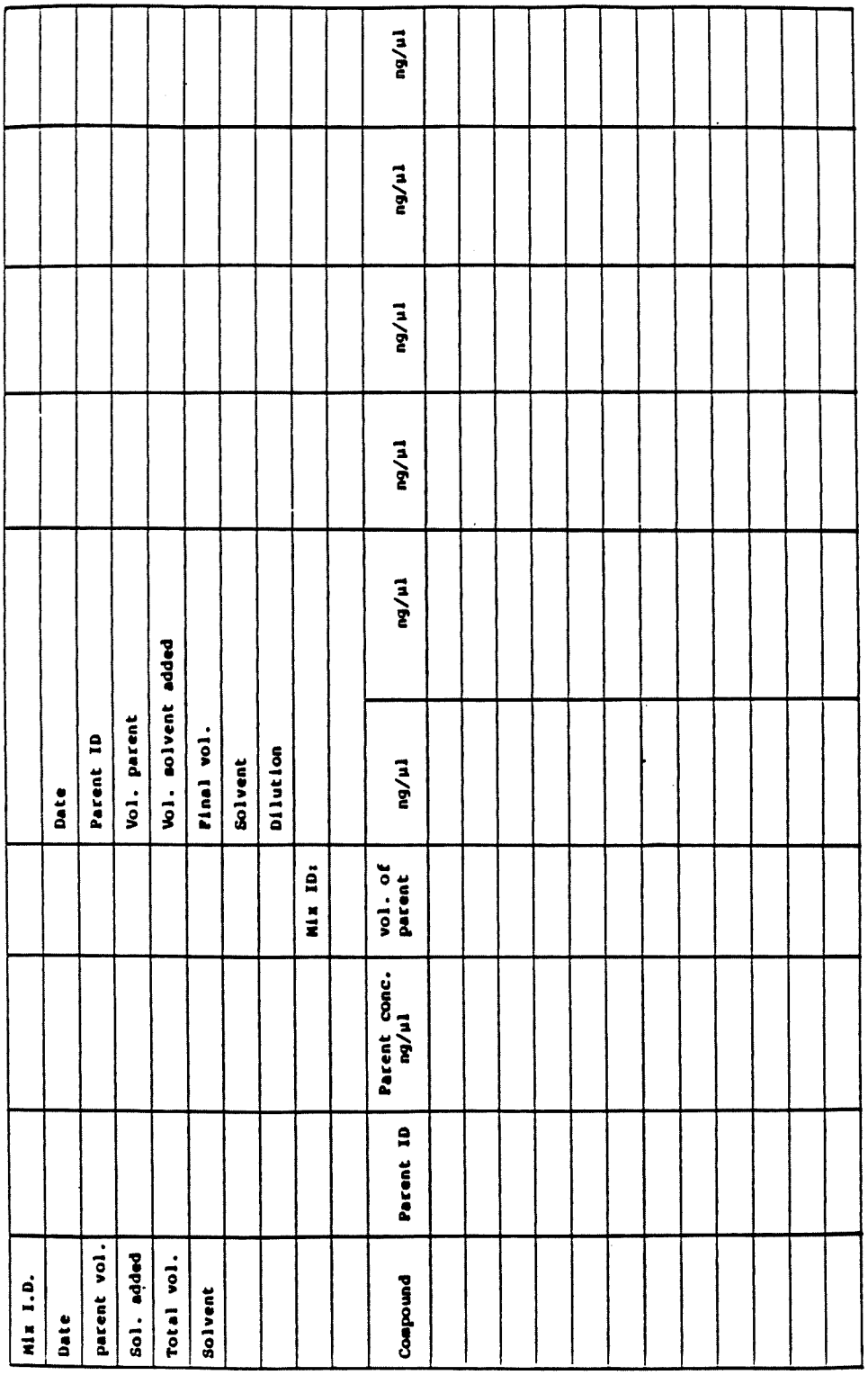
Erco's primary sources of standard reference materials for calibrations and calibration checks are the U.S. EPA and National Bureau of Standards (NBS) repositories. Secondary sources are reliable commercial supply houses such as Supelco, Aldrich, and Chem Service. New standards are routinely checked against known standards that are traceable to EPA or NBS reference materials, if available. All of Erco's laboratories maintain a standards preparation logbook in which all pertinent information regarding the source and preparation of each analytical standard is recorded. An example is shown in figure 6-1.

Reagents used in the preparation of laboratory control spike and laboratory control spike duplicate solutions and in surrogate standard spiking solutions are obtained directly from the U.S. EPA or are validated using EPA-traceable standards. QC check samples are requested and received on a frequent basis from EPA Environmental Monitoring and Support Laboratory (EMSL) in Cincinnati, Ohio.

Reagents that are used in large quantities are analyzed for purity at Erco's laboratories prior to purchase and according to lot number.

Parent	Compound	Date	Chemist	Parent (stock) Concentration (ng/µl)	Dilution parent. stock	Vol. Parent Solution	Vol. Solvent Added	Final Vol.	Solvent	Conc. ng/ul

Figure 6-1. Standards Log Sheet.



Mix I.D.																							
Date											Date												
parent vol.											Parent ID												
Sol. added											Vol. parent												
Total vol.											Vol. solvent added												
Solvent											Final vol.												
											Solvent												
											Dilution												
											Mix ID:												
Compound	Parent ID	Parent conc. ng/µl	vol. of parent	ng/µl	ng/µl	ng/µl	ng/µl	ng/µl	ng/µl	ng/µl													

Figure 6-1. Continued.

6.4 Instrumentation

The Erco instrumentation that will be employed for this project is described in this section.

The GC/MS systems that will be used for semivolatile organic analyses are:

- One Finnigan MAT 4600/Super INCOS GC/LC/MS/DS;
- One Finnigan MAT 4500/Super INCOS GC/MS/DS;
- One Finnigan MA 4521/INCOS GC/MS/DS;
- One Hewlett Packard 7970B magnetic tape unit; and
- One Cipher magnetic tape unit.

The GC/MS systems that will be used for volatile organic analyses are:

- Three Hewlett Packard 5996 GC/MS systems with HP RTE-6 data systems and Tekmar LSC-2 purge and traps with Tekmar ALS automatic sampler; and
- One Hewlett Packard 5985 GC/MS with HP RTE-6 data system and Tekmar LSC-2 purge and traps with a Tekmar ALS automatic sampler.

The GC systems that will be used for aromatic volatile organic analyses are:

- One Varian 3700 with photoionization, Hall electrolytic conductivity and flame ionization detectors, and Tekmar purge and traps with Tekmar automatic sampler;
- One Perkin Elmer 3920 with photoionization, Hall electrolytic conductivity and flame ionization detectors, and Tekmar 4000 automatic heated sampler module; and
- Two Hewlett-Packard 5890 GC systems with photoionization, Hall electrolytic conductivity detectors, and Tekmar purge and traps.

The GC systems that will be used for pesticides analyses are:

- One Hewlett Packard 5890 GC system with an electron capture detector; and

- Two Hewlett Packard 5890 GC systems with a flame photometric detector in phosphorous mode.

Additional systems include:

- One Jarrell-Ash 9000 inductively coupled argon plasma emission spectrometer;
- One Perkin Elmer 3030 Zeeman atomic absorption spectrophotometer with HGA-600 graphite furnace;
- One Fisher mercury analyzer; and
- Other various inorganics analyzers.

7. ANALYTICAL PROCEDURES

In accordance with the objectives of this QAPP, specific procedures for the analyses of Appendix IX VOCs and SVCs and volatile aromatic compounds will be performed. A list of the VOCs and SVCs indicated is provided, with detection limits, in tables 7-1 and 7-2. Tables 7-3 through 7-7 list compounds and reporting limits for all other Appendix IX constituents. Table 7-8 lists the volatile aromatic compounds and their reporting limits. Tables 7-9 and 7-10 list the compounds and reporting limits for methods 8240 and 8270, respectively.

Samples will be prepared according to the methods referenced in Tables 4-1 and 4-2. For semivolatile organics analyses, water samples will undergo solvent extraction using a separatory funnel and concentration by Kuderna-Danish (KD) apparatus. A liquid/liquid extraction procedure will be used as an alternative if the sample matrix requires use of this method. Volatile organics analyses in water require no sample preparation as the volatile compounds are purged from the sample and desorbed into the chromatographic column by the purge-and-trap apparatus. Gel permeation chromatography (GPC) will be used for the cleanup of soil extracts for pesticides analysis. Other cleanups, such as florisil and copper, will be used as necessary.

All SOPs are on file at Erco and are available for inspection at the time of a site audit. Any SOPs will be made available to the U.S. EPA for evaluation, if required. All of Enseco's SOPs are considered confidential business information.

Dioxin and furan analyses will be performed at Enseco - California Analytical Laboratory in West Sacramento, California. Sulfide analyses will be performed at Enseco - Rocky Mountain Analytical Laboratory in Arvada, Colorado. All other work will be performed at Erco. However, if it becomes necessary to have another Enseco laboratory provide assistance, the project manager at Hatcher-Sayre, Inc. will be contacted for approval.

For Appendix IX compounds that are not included in the SW-846 list of compounds, an authentic standard containing all of these compounds will be prepared and analyzed prior to all sample analyses and following each successive SW-846 continuing calibration. The standard will be in the midrange of the working calibration curve.

Table 7-1. Appendix IX volatile organic compounds and Erco reporting limits for water

Compound	Reporting Limit[a] Water (µg/L)
Chloromethane	10
Bromomethane	10
Dichlorodifluoromethane	10
Vinyl chloride	10
Acetonitrile	100
Chloroethane	10
Iodomethane	5.0
Dichloromethane	5.0
Acrolein	100
Acetone	25
Acrylonitrile	100
Carbon disulfide	5.0
Ethyl cyanide	5.0
Trichlorofluoromethane	5.0
1,1-Dichloroethene	5.0
Allyl chloride	5.0
1,1-Dichloroethane	5.0
1,2-Dichloroethene (total)	5.0
Chloroform	5.0
1,2-Dichloroethane	5.0
Methyl ethyl ketone (MEK)	25
Methacrylonitrile	5.0
Dibromomethane	5.0
1,1,1-Trichloroethane	5.0
Isobutyl alcohol	25
1,4-Dioxane	b
Carbon tetrachloride	5.0
Vinyl acetate	10
Bromodichloromethane	5.0
Chloroprene	5.0
1,2-Dichloropropane	5.0
trans-1,3-Dichloropropene	5.0
Trichloroethene	5.0
Dibromochloromethane	5.0
1,1,2-Trichloroethane	5.0
Benzene	5.0
cis-1,3-Dichloropropene	5.0
1,2-Dibromoethane	5.0
Bromoform	5.0

[a]Specific quantitation limits are highly matrix-dependent. The quantitation limits are provided for guidance and may not always be achievable.

[b]Reporting limit has not yet been determined.

Table 7-1. Continued

Compound	Reporting Limit[a] Water (µg/L)
1,1,1,2-Tetrachloroethane	5.0
4-Methyl-2-pentanone	10
2-Hexanone	10
1,2,3-Trichloropropane	5.0
trans-1,4-Dichloro-2-butene	5.0
1,1,2,2-Tetrachloroethane	5.0
Tetrachloroethene	5.0
Toluene[c]	5.0
Chlorobenzene	5.0
Ethyl benzene	5.0
1,2-Dibromo-3-chloropropane	5.0
Styrene	5.0
Xylenes (total)	5.0
Methyl methacrylate	b
Pyridine	b
Ethyl methacrylate	b
2-Picoline	b

[a]Specific quantitation limits are highly matrix-dependent. The quantitation limits are provided for guidance and may not always be achievable.

[b]Reporting limit has not yet been determined.

[c]High concentrations of toluene may be present in many samples. Diluted sample analyses may be required in order to detect toluene concentrations within a working range. In such cases, the samples will also be analyzed at the minimum dilution that will not cause instrument failure so that other components may be reported at their minimum reporting limits.

Table 7-2. Appendix IX semivolatile organic compounds and Erco reporting limits for water

Compound	Reporting Limit[a] Water (µg/L)
N-Nitrosodimethylamine	10
N-Nitrosomethylethylamine	10
Methyl methanesulfonate	10
N-Nitrosodiethylamine	10
Ethyl methanesulfonate	10
Pentachloroethane	10
Aniline	10
Phenol	10
bis(2-Chloroethyl)ether	10
2-Chlorophenol	10
1,3-Dichlorobenzene	10
1,4-Dichlorobenzene	10
Benzyl alcohol	20
1,2-Dichlorobenzene	10
bis(2-Chloroisopropyl)ether	10
N-Nitroso-di-n-propylamine	10
o-Cresol	10
Acetophenone	10
N-Nitrosopyrrolidine	10
Hexachloroethane	10
N-Nitrosomorpholine	10
o-Toluidine	10
m & p-Cresol(s)	10
Nitrobenzene	10
N-Nitrosopiperidine	10
a,a-Dimethylphenethylamine	10
Isophorone	10
2-Nitrophenol	10
2,4-Dimethylphenol	10
bis(2-Chloroethoxy)methane	10
2,4-Dichlorophenol	10
1,2,4-Trichlorobenzene	10
Naphthalene	10
2,6-Dichlorophenol	10
4-Chloroaniline	20
Hexachloropropene	20
Hexachlorobutadiene	10
p-Phenylenediamine	b

[a]Specific quantitation limits are highly matrix-dependent. The quantitation limits are provided for guidance and may not always be achievable.

[b]Reporting limit has not yet been determined.

Table 7-2. Continued

Compound	Reporting Limit[a] Water (µg/L)
N-Nitrosodi-n-butylamine	10
Safrole	10
4-Chloro-3-methylphenol	20
2-Methylnaphthalene	10
1,2,4,5-Tetrachlorobenzene	10
Hexachlorocyclopentadiene	10
2,4,6-Trichlorophenol	10
2,4,5-Trichlorophenol	50
2-Chloronaphthalene	10
Isosafrole	20
2-Nitroaniline	50
1,4-Naphthoquinone	10
Acenaphthylene	10
1,3-Dinitrobenzene	10
Dimethyl phthalate	10
2,6-Dinitrotoluene	10
Acenaphthene	10
3-Nitroaniline	50
2,4-Dinitrophenol	50
Dibenzofuran	10
Pentachlorobenzene	10
2-Naphthalenamine	10
2,4-Dinitrotoluene	10
4-Nitrophenol	50
1-Naphthylamine	10
Fluorene	10
4-Chlorophenyl phenyl ether	10
5-Nitro-o-toluidine	10
Diethyl phthalate	10
4-Nitroaniline	50
4,6-Dinitro-o-cresol	50
N-Nitrosodiphenylamine	10
Diphenylamine	10
4-Bromophenyl phenyl ether	10
sym-Trinitrobenzene	10
Phenacetin	10
Hexachlorobenzene	10
4-Aminobiphenyl	10
Pentachlorophenol	50

[a]Specific quantitation limits are highly matrix-dependent. The quantitation limits are provided for guidance and may not always be achievable.

Table 7-2. Continued

Compound	Reporting Limit[a] Water (µg/L)
Pentachloronitrobenzene	50
Phenanthrene	10
Anthracene	10
2-sec-Butyl-4,6-dinitrophenol	10
Di-n-butyl phthalate	10
4-Nitroquinoline-1-oxide	b
Methapyrilene	10
Fluoranthene	10
Pyrene	10
Aramite	10
p-Dimethylaminoazobenzene	10
3,3'-Dimethylbenzidine	10
Butyl benzyl phthalate	10
2-Acetylaminofluorene	10
Benzo(a)anthracene	10
Chrysene	10
3,3'-Dichlorobenzidine	20
bis(2-Ethylhexyl)phthalate	10
Di-n-octyl phthalate	10
Benzo(b)fluoranthene	10
Benzo(k)fluoranthene	10
7,12-Dimethylbenzanthracene	10
Benzo(a)pyrene	10
Hexachlorophene	b
3-Methylcholanthrene	20
Indeno(1,2,3-c,d)pyrene	10
Dibenzo(a,h)anthracene	10
Benzo(g,h,i)perylene	10
Pronamide	20
2,3,4,6-Tetrachlorophenol	20
Famphur	b

[a]Specific quantitation limits are highly matrix-dependent. The quantitation limits are provided for guidance and may not always be achievable.

[b]Reporting limit has not yet been determined.

Table 7-3. Appendix IX organochlorine and organophosphorous pesticides and Erco reporting limits

Compound	Reporting Limit[a] (in µg/L)
alpha-BHC	0.050
beta-BHC	0.050
delta-BHC	0.050
gamma-BHC (Lindane)	0.050
Heptachlor	0.050
Aldrin	0.050
Heptachlor epoxide	0.050
Endosulfan I	0.050
Dieldrin	0.10
4,4'-DDE	0.10
Endrin	0.10
Endosulfan II	0.10
4,4'-DDD	0.10
Endosulfan sulfate	0.10
4,4'-DDT	0.10
Endrin aldehyde	0.10
Methoxychlor	0.50
Chlordane	0.50
Toxaphene	1.0
Aroclor-1016	0.50
Aroclor-1221	0.50
Aroclor-1232	0.50
Aroclor-1242	0.50
Aroclor-1248	0.50
Aroclor-1254	1.0
Aroclor-1260	1.0
Chlorobenzilate	0.50
Diallate	1.5
Isodrin	0.050
Kepone	b
Phorate	1.0
Disulfoton	1.0
Dimethoate	1.0
Methyl parathion	1.0
Ethyl parathion	1.0
Famphur	1.0
Sulfotepp	1.0
Thionazin	1.0
o,o,o-Triethylphosphorothi	1.0

[a]Specific quantitation limits are highly matrix-dependent. The quantitation limits are provided for guidance and may not always be achievable.

[b]Reporting limit has not yet been determined.

Table 7-4. Appendix IX total metals compounds and Erco reporting limits

Compound	Reporting Limit[a] (in mg/L)
Antimony	0.05
Arsenic	0.003
Barium	0.005
Beryllium	0.001
Cadmium	0.005
Chromium	0.01
Cobalt	0.01
Copper	0.006
Lead	0.005
Mercury	0.0002
Nickel	0.04
Selenium	0.005
Silver	0.005
Thallium	0.004
Tin	0.03
Vanadium	0.01
Zinc	0.01

[a]Specific quantitation limits are highly matrix-dependent. The quantitation limits are provided for guidance and may not always be achievable.

Table 7-5. Appendix IX inorganic compounds and Erco reporting limits

Compound	Reporting Limit[a] (in mg/L)
Cyanide, total	0.01
Sulfide[b]	1.0

[a]Specific quantitation limits are highly matrix-dependent. The quantitation limits are provided for guidance and may not always be achievable.

[b]This analysis will be performed at Enseco - Rocky Mountain Analytical Laboratory.

Table 7-6. Appendix IX herbicide compounds and Erco reporting limits

Compound	Reporting Limit[a] (in µg/L)
2,4-D	0.25
2,4,5-TP (Silvex)	0.050
2,4,5-T	0.050
Dinoseb	b

[a]Specific quantitation limits are highly matrix-dependent. The quantitation limits are provided for guidance and may not always be achievable.

[b]Reporting limit has not yet been determined.

Table 7-7. Appendix IX dioxin and furan compounds and Erco reporting limits

Compound	Reporting Limit[a] (in ng/L)
TCDFs (total)	1
PeCDFs (total)	1
HeCDFs (total)	1
TCDDs (total)	1
PeCDDs (total)	1
HeCDDs (total)	1
2,3,7,8-TCDD	5

[a]Specific quantitation limits are highly matrix-dependent. The quantitation limits are provided for guidance and may not always be achievable.

Table 7-8. Aromatic volatile organics

Compound	Reporting Limits[a]	
	Water (µg/L)	Soil (µg/kg)
Benzene	1.0	2.0
Toluene[b]	1.0	2.0
Ethylbenzene	1.0	2.0
Xylene (total)	1.0	2.0

[a]Specific quantitation limits are highly matrix-dependent. The quantitation limits are provided for guidance and may not always be achievable.

[b]High concentrations of toluene may be present in many samples. Diluted sample analyses may be required in order to detect toluene concentrations within a working range. In such cases, the samples will also be analyzed at the minimum dilution that will not cause instrument failure so that other components may be reported at their minimum reporting limits.

Table 7-9. Method 8240 volatile organic compounds and Erco reporting limits for soil and water

Compound	Reporting Limits[a] Water (µg/L)	Soil (µg/kg)
Chloromethane	10	20
Bromomethane	10	20
Vinyl chloride	10	20
Chloroethane	10	20
Dichloromethane	25	40
Acetone	25	40
Carbon disulfide	5.0	10
1,1-Dichloroethene	5.0	10
1,1-Dichloroethane	5.0	10
1,2-Dichloroethene (total)	5.0	10
Chloroform	5.0	10
1,2-Dichloroethane	5.0	10
Methyl ethyl ketone (MEK)	25	40
1,1,1-Trichloroethane	5.0	10
Carbon tetrachloride	5.0	10
Vinyl acetate	10	20
Bromodichloromethane	5.0	10
1,2-Dichloropropane	5.0	10
trans-1,3-Dichloropropene	5.0	10
Trichloroethene	5.0	10
Dibromochloromethane	5.0	10
1,1,2-Trichloroethane	5.0	10
Benzene	5.0	10
cis-1,3-Dichloropropene	5.0	10
Bromoform	5.0	10
1,1,1,2-Tetrachloroethane	5.0	10
4-Methyl-2-pentanone	10	20
2-Hexanone	10	20
1,1,2,2-Tetrachloroethane	5.0	10
Tetrachloroethene	5.0	10
Toluene[b]	5.0	10
Chlorobenzene	5.0	10
Ethyl benzene	5.0	10
Styrene	5.0	10
Xylenes (total)	5.0	10

[a]Specific quantitation limits are highly matrix-dependent. The quantitation limits are provided for guidance and may not always be achievable.

[b]High concentrations of toluene may be present in many samples. Diluted sample analyses may be required in order to detect toluene concentrations within a working range. In such cases, the samples will also be analyzed at the minimum dilution that will not cause instrument failure so that other components may be reported at their minimum reporting limits.

Table 7-10. Method 8270 semivolatile organic compounds and Erco reporting limits for water

Compound	Reporting Limits[a] Water (µg/L)	Soil (µg/kg)
Phenol	10	330
bis(2-Chloroethyl)ether	10	330
2-Chlorophenol	10	330
1,3-Dichlorobenzene	10	330
1,4-Dichlorobenzene	10	330
Benzyl alcohol	20	660
1,2-Dichlorobenzene	10	330
bis(2-Chloroisopropyl)ether	10	330
N-Nitroso-di-n-propylamine	10	330
2-Methylphenol	10	330
4-Methylphenol	10	330
Hexachloroethane	10	330
Nitrobenzene	10	330
Isophorone	10	330
2-Nitrophenol	10	330
2,4-Dimethylphenol	10	330
bis(2-Chloroethoxy)methane	10	330
2,4-Dichlorophenol	10	330
1,2,4-Trichlorobenzene	10	330
Naphthalene	10	330
4-Chloroaniline	20	660
Hexachlorobutadiene	10	330
4-Chloro-3-methylphenol	20	660
2-Methylnaphthalene	10	330
Hexachlorocyclopentadiene	10	330
2,4,6-Trichlorophenol	10	330
2,4,5-Trichlorophenol	50	1,600
2-Chloronaphthalene	10	330
2-Nitroaniline	50	1,600
Acenaphthylene	10	330
Dimethyl phthalate	10	330
2,6-Dinitrotoluene	10	330
Acenaphthene	10	330
3-Nitroaniline	50	1,600
2,4-Dinitrophenol	50	1,600
Dibenzofuran	10	330
2,4-Dinitrotoluene	10	330
4-Nitrophenol	50	1,600

[a]Specific quantitation limits are highly matrix-dependent. The quantitation limits are provided for guidance and may not always be achievable.

[b]Reporting limit has not yet been determined.

Table 7-10. Continued

Compound	Reporting Limits[a] Water (µg/L)	Soil (µg/kg)
Fluorene	10	330
4-Chlorophenyl phenyl ether	10	330
Diethyl phthalate	10	330
4-Nitroaniline	50	1,600
4,6-Dinitro-2-methylphenol	50	1,600
N-Nitrosodiphenylamine[c]	10	330
4-Bromophenyl phenyl ether	10	330
Hexachlorobenzene	10	330
Pentachlorophenol	50	1,600
Phenanthrene	10	330
Anthracene	10	330
Di-n-butyl phthalate	10	330
Fluoranthene	10	330
Pyrene	10	330
Butyl benzyl phthalate	10	330
Benzo(a)anthracene	10	330
Chrysene	10	330
3,3'-Dichlorobenzidine	20	660
bis(2-Ethylhexyl)phthalate	10	330
Di-n-octyl phthalate	10	330
Benzo(b)fluoranthene	10	330
Benzo(k)fluoranthene	10	330
Benzo(a)pyrene	10	330
Indeno(1,2,3-c,d)pyrene	10	330
Dibenzo(a,h)anthracene	10	330
Benzo(g,h,i)perylene	10	330

[a]Specific quantitation limits are highly matrix-dependent. The quantitation limits are provided for guidance and may not always be achievable.

[c]Quantitated as diphenylamine.

8. DATA REDUCTION, VALIDATION, AND REPORTING

8.1 Data Reduction and Validation

All analytical data generated are extensively checked for accuracy and completeness. The data validation process consists of data generation, reduction, and three levels of review as described below.

The analyst who generates the analytical data has the prime responsibility for the correctness and completeness of the data. All data are generated and reduced following protocols specified in laboratory SOPs. Each analyst reviews the quality of his work based on an established set of guidelines. The analyst reviews the data package to ensure that:

- sample preparation information is correct and complete;
- analysis information is correct and complete;
- the appropriate SOPs have been followed;
- analytical results are correct and complete;
- QC samples are within established control limits;
- special sample preparation and analytical requirements have been met; and
- documentation is complete (e.g., all anomalies in the preparation and analysis have been documented; out-of-control forms are complete, if required; holding times are documented, etc.)

The data reduction and validation steps are documented, signed and dated by the analyst. This initial review step, performed by the analyst, is designated as the level 1 review. The analyst then passes the data package to an independent reviewer who performs a level 2 review.

The level 2 review is performed by a data review specialist whose function is to provide an independent review of the data package. This review

is also conducted according to an established set of guidelines and is structured to ensure that:

- calibration data are scientifically sound, appropriate to the method, and completely documented;
- QC samples are within established guidelines;
- qualitative identification of sample components is correct;
- quantitative results are correct;
- documentation is complete and correct (e.g., anomalies in the preparation and analysis have been documented; out-of-control forms are complete, if required; holding times are documented, etc.);
- the data are ready for incorporation into the final report; and
- the data package is complete and ready for data archiving.

The level 2 review is structured so that all calibration data and QC sample results are reviewed and all of the analytical results from 10% of the samples are checked back to the benchsheet. If no problems are found with the data package, the review is complete. If any problems are found with the data package, an additional 10% of the samples are checked to the benchsheet. The process continues until no errors are found or until the data package has been reviewed in its entirety.

An important element of the level 2 review is the documentation of any errors that have been identified and corrected during the review process. Enseco believes that the data package submitted by the analyst for level 2 review should be free of errors. Any errors that are found are documented and transmitted to the appropriate supervisor. The cause of each error is then addressed with additional training or clarification of procedures to ensure that quality data will be generated at the bench.

The level 2 data review is also documented and the signature of the reviewer and the date of review recorded. The reviewed data are then approved for release and a final report is prepared.

Before the report is released to the client, the client manager reviews the report to ensure that the data meet the overall objectives of the client, as understood by the client manager. This review is labeled as the level 3 review.

In addition, the divisional QA department randomly audits 5% of all projects reported. The QA audit includes verifying that holding times have been met; calibration checks are adequate; qualitative and quantitative results are correct; documentation is complete; and QC results are complete and accurate. During the review, the QA department checks the data from 20% of the samples back to the benchsheet. If no problems are found with the data package, the review is complete. If any problems are found with the data package, an additional 10% of the samples are checked back to the benchsheet. The process continues until no errors are found or until the data package has been reviewed in its entirety.

8.2 Data Reporting

Data will be reported containing the items specified in the EPA SOW 887. A sample data summary package will accompany the data for each sample delivery group (SDG). Each sample data summary package will contain a case narrative and the sample data (arranged by fraction). Sample data will include target compound results, tentatively identified compound results, surrogate spike analysis results, matrix spike/matrix spike duplicate (MS/MSD) results, blank data results, and internal standard area results. Also included in each data package will be chain-of-custody information, raw data (including chromatograms), mass spectra and tables, standards data, BFB and DFTPP tune data, and blank data. Copies of all worksheets; data system printouts from GC, GC/MS, GPC, ICP, graphite furnace, and atomic absorption instruments; forms from sample preparation and analysis; telephone logbook pages; personal logbook pages; and any case-specific notes will also be included.

All data will be reported using conventional CLP quality control data and qualifiers including 'J' flags for results found at concentrations less than

the reporting limit. The actual value found (along with the 'J' flag) will be reported.

8.3 Data Archival

A complete copy of each data package submitted will be archived at the laboratory. This will also include a case file purge which consists of all laboratory records received or generated for a specific case that were not submitted as deliverables. The data package and case file purge will be archived in a secure area which may be accessed only by personnel of the document control and quality assurance departments. All files removed from the area will be signed out by the person removing them in a data archival logbook.

9. INTERNAL QUALITY CONTROL CHECKS

9.1 Field QC Checks

Field QC checks will be utilized during this investigation through the use of the following items.

- Trip Blanks - One trip blank per cooler will be run for every 20 samples for the analysis of volatile organics. Trip blanks will be prepared by the laboratory with deionized water, preserved with hydrochloric acid (HCl), transported to the site, handled like a sample, and returned to the laboratory for analysis. Trip blanks are not opened until they are received by the laboratory for analysis.

- Field Blanks - Field blanks are prepared in the field by routing deionized water through the decontaminated sample collection device and collecting it in the sampler container. The samples are preserved and returned to the laboratory for volatile organics analyses. One field blank will be collected for every 10 samples.

- Field Duplicates - Field duplicates are two sets of samples from a single sample location that are prepared and labeled with unique sample numbers and submitted to the laboratory to determine analytical precision and sample representativeness. One field duplicate will be collected for every 10 samples.

- Matrix Spikes and Matrix Spike Duplicates - Matrix spike and matrix spike duplicate samples will be analyzed at a frequency of 1 MS/MSD for every 20 samples. Extra samples will be collected in the field as required.

9.2 Laboratory QC Checks

All QC procedures employed at Erco will be, at a minimum, those outlined in U.S. EPA Test Methods for Evaluating Solid Waste, SW-846 (third edition, November 1986). General QC protocols for trace organic analyses include the following items.

- Field blanks, when applicable, are used to detect contamination introduced during sampling, shipping, and handling.

- A minimum of one procedural blank (i.e., method blank) is run for every 20 samples analyzed to detect contamination during analysis.
- One laboratory control spike and one laboratory control spike duplicate will be run for every 20 samples to determine recovery and precision.
- Surrogate standards and internal standards will be run to quantitate results, determine recoveries, and to account for sample-to-sample variation. See table 9-1 for a list of surrogates to be used.
- Calibration of GCs and GC/MS's will be determined according to the appropriate EPA CLP methods.
- Tuning of GC/MS systems every 12 hours will meet EPA criteria using BFB for volatile organics analysis, and DFTPP for semivolatile analysis, as described in section 8.0.
- Routine analysis of performance evaluation samples and blind spikes will be used to document method accuracy and precision.
- Multilevel initial calibrations of instruments will be made to establish calibration curves.
- Continuing calibration standards will be run at least once every 12 hours of instrumental analysis for accurate quantitation. Recalibration may result if these do not meet criteria.

9.3 Control Limits

The control limits that will be used to determine accuracy and precision are defined in U.S. EPA "Methods for Evaluating Solid Waste" (third edition, November 1986). For analytes listed in Appendix IX but not in SW-846, control limits will be established using method validation data. The limits will be ±3 standard deviations (SD) from the mean for accuracy. The limits for precision, expressed through RPD, will be +3 SD from the mean.

Table 9-1. Surrogates used for appendix 9 analysis

EPA Method	Compound
8080	
8140	Diazinon Malathion Parathion Ethion
8150	2,4-DB
8240	1,2-Dichloroethane-d_4 Toluene-d_8 4-Bromofluorobenzene
8270	Nitrobenzene-d_5 2-Fluorobiphenyl Terphenyl-d_{14} Phenol-d_5 2-Fluorophenol 2,4,6-Tribromophenol
613 (Dioxins and furans)	37Cl-2,3,7,8-TCDD

Table 10-1. Erco systems audits -- January 1989 to June 1990

	Auditor	Date
1)	U.S. EPA Region I TechLaw, Lockheed Inorganic CLP audit	February 1989
2)	Florida Department of Health and Rehabilitative Services Laboratory certification audit	May 1989
3)	Edward Maser Pennsylvania Department of Environmental Regulation Laboratory certification audit	October 1989
4)	Martin Marietta Energy Systems	December 1989
5)	New York Department of Health ELAP certification	March 1990
6)	New Jersey DEP Project-specific audit	April 1990
7)	U.S. EPA Region II Army Corps of Engineer project	April 1990
8)	U.S. EPA Region I TechLaw Organic CLP audit	April 1990
9)	New York State Department of Environmental Conservation Contract audit	June 1990
10)	U.S. EPA Region V Appendix IX approval Site-specific audit	June 1990

Table 10-2. Erco performance evaluations -- January 1989 to January 1990

	Agency/Program	Parameters	Date
1)	U.S. EPA CLP QB2, FY-89	Inorganics	January 1989
2)	U.S. EPA Water Pollution Study WP-022	VOA, pesticides, PCBs, metals, inorganics	March 1989
3)	New York DOH Nonpotable Water	Semivolatile organics, VOA, inorganics, metals, PCBs, pesticides	March 1989
4)	U.S. EPA Water Supply Study WS-024	VOA, metals, inorganics, pesticides, herbicides	May 1989
5)	New York DOH Potable Water Study	Trihalomethanes, metals, pesticides, herbicides, inorganics	May 1989
6)	New York DOH Nonpotable Water	Semivolatile organics, VOA, inorganics, metals, PCBs, pesticides	August 1989
7)	U.S. EPA CLP QB4, FY-89	Semivolatile organics, VOA, pesticides	September 1989
8)	U.S. EPA Water Pollution Study WP-023	VOA, pesticides, PCBs, metals, inorganics	September 1989
9)	U.S. EPA Water Supply Study WS-025	VOA, metals, inorganics, pesticides, herbicides	November 1989
10)	New York DOH Potable Water Study	Trihalomethanes, metals, pesticides, herbicides, inorganics	November 1989
11)	U.S. EPA CLP QB1, FY-90	Semivolatile organics, VOA, pesticides	December 1989
12)	New York State Department of Environmental Conservation	September 1989 ASP Contract Pre-award PE	February 1990

Table 10-3. EPA quarterly blind performance evaluation studies

EPA ID	Date	Raw Score (%)
1) QB4, FY-84, Case #3061	November 1984	82
2) QB1, FY-85, Case #3287	February 1985	77
3) QB2, FY-85, Case #3821	May 1985	96
4) QB3, FY-85, Case #4183	August 1985	90
5) QB4, FY-85, Case #4604	December 1985	93
6) QB1, FY-86, Case #5076	March 1986	100
7) QB2, FY-86, Case #5423	May 1986	99
8) QB3, FY-86, Case #5772	August 1986	94
9) QB4, FY-86, Case #6076	November 1986	83.7
10) QB1, FY-87, Case #6437	January 1987	96
11) QB2, FY-87, Case #6666	May 1987	96
12) QB3, FY-87, Case #7144	July 1987	100
13) QB4, FY-87, Case #7760	December 1987	97
14) QB3, FY-88, Case #9300	July 1988	86
15) QB3, FY-88, Case #9302	June 1988	93
16) QB4, FY-88, Case #10015	September 1988	96
17) QB1, FY-89, Case #10584	December 1988	67
18) QB2, FY-89, Case #11273	March 1989	99
19) QB4, FY-89, Case #12326	September 1989	53
20) QB1, FY-90, Case #12895	December 1989	73
21) QB2, FY-90, Case #13456	January 1990	96

ENSECO INCORPORATED

QA WALKTHROUGH

GC LAB--PESTICIDES

		Yes/No/Comments
1)	Are current SOP's available for all personnel in the area?	
2)	Have new personnel read the SOP's for this area?	
3)	Are DCS or SCS analyzed with each set of samples as required in M-EQA-002?	
4)	Are method blanks analyzed with every batch of samples?	
5)	Are QC Samples analyzed before associated analytical samples?	
6)	Are results of QC samples verified to determine if QC criteria have been met before sample analysis begins?	
7)	Are QC results which are outside of acceptance limits checked for error? What procedures are followed?	
8)	Are all QC results, both acceptable and unacceptable entered into LIMS?	
9)	Are anomalies documented and reported to QA as required?	
10)	Are corrective actions taken as necessary and documented and samples reprepped/reanalyzed?	
11)	Is data acceptance based on results of SCS or DCS unless otherwise required by the client?	
12)	If holding times are exceeded, are proper procedures followed to notify QA and the client?	
13)	Is the balance located away from drafts and areas subject to rapid temperature changes?	
14)	Is the balance calibration checked daily (i.e. any day balance used), and recorded?	

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Figure 10-1. Laboratory Audit Form.

11. PREVENTATIVE MAINTENANCE

Erco maintains a complete inventory of replacement parts needed for preventative maintenance and spare parts that routinely need replacement (e.g., ferrules, traps, gauges, detectors, filaments, etc.) If an instrument fails, the problem will be diagnosed as quickly as possible and either replacement parts will be ordered or a service call will be placed with the manufacturer. Service contracts are in effect for the data systems controlling the operation of the GC/MS systems.

Preventative maintenance schedules for analytical instrumentation are included in Erco's laboratory manuals and are shown in figure 11-1.

PREVENTIVE MAINTENANCE CHECKS

ITEM	SERVICE INTERVAL[1]						SERVICE LEVEL
	Dy	Wk	Mo	Qu	Bi	An	
HP 5840A GC							
• Septa		X					Inspect and replace as required.
• Injection port liner		X					Clean or replace.
• Carrier gas filter				X			Replace.
• Capillary column		X			X		Clip or replace as required.
HP 5985 MS							
• Ion gas tube	X						Operate Degas mode for 30 sec.
• Ion source filament				X			Replace as needed.
• Ion source					X		Inspect and clean.
• Electron multiplier					X		Replace as required.
• Teflon seal in direct introduction inlet				X			Replace.
• Vacuum pumps					X		Inspect belt and change oil.
HP 7900 Disc Drive					X		Service contract P.M.
Tektronix hardcopy unit			X				Clean and inspect.
					X		Service contract P.M.
Finnigan 9610 GC							
• Septa		X					Inspect and replace as required.
• Injection port liner		X					Clean or replace.
• Carrier gas filter				X			Replace.
• Capillary column		X			X		Clip or replace as required.
Printronix Printer							
• Interior			X				Clean and inspect.
• Printer ribbon			X				Change as needed.
• Preventive maintenance					X		
Finnigan 4530 MS							
• Quadrupole mass analyzer					X		Clean rod assembly.
• Ion source				X			Clean and inspect.
• Cal gas assembly					X		Inspect.
• Ion source filament				X			Replace as needed.
• Electron multiplier					X		Replace as needed.
• Vacuum pumps				X			Inspect and change oil.
Control Data CMD Disk Drive				X			Service contract preventive maintenance.
• Air filters				X			
• Power supply outputs					X		
• Actuator assembly					X		

[1]Da = daily; We = weekly; Mo = monthly; Qu = quarterly; Bi = biannually; An = annually.

Figure 11-1. Maintenance for GC/MS Systems.

12. SPECIFIC ROUTINE PROCEDURES USED TO ASSESS DATA QUALITY AND DETERMINE DETECTION LIMITS

By following all of the procedures outlined in this QAPP and by thoroughly documenting all work that is performed, Erco will closely monitor data precision, accuracy, and completeness. Validity of detection limits is also assured.

12.1 Data Quality

For this project, the methods to determine precision and accuracy, and their acceptability, are well-defined in each of the specific methods.

12.2 Precision

Precision is frequently determined by the comparison of duplicates and laboratory control spike duplicates. Duplicates result from an original sample that has been split for identical analyses. The RPD of a result is commonly used in estimating precision.

The RPD of duplicate samples and laboratory control spike duplicates will be used to estimate the precision. The following equation will be used to determine this.

- $$RPD = \frac{|D_1 - D_2|}{(D_1 + D_2)/2} \times 100$$

  where:

  RPD = relative percent difference;
  D_1 = first sample value; and
  D_2 = second sample value (duplicate).

12.3 Accuracy

The determination of accuracy of a measurement requires a knowledge of the true or accepted value for the analyte being measured. Accuracy will be calculated in terms of percent recovery in the following equation.

- Percent recovery = $100 \frac{\bar{X}}{T}$

where:

$\bar{X}$ = mean of observed value(s) for measurement(s); and
T = "true" value.

12.4 Analytical Completeness

Determining whether a data base is complete or incomplete can be quite difficult. To be considered complete, the data set must contain all QC check analyses verifying precision and accuracy for all of the analytical protocol. Less obvious is whether that data are sufficient to achieve the goals of the project. All data are reviewed in terms of goals in order to determine if the data base is sufficient.

Whenever possible, the percent completeness for each set of samples is calculated as in the following equation.

- $\text{Completeness} = \frac{\text{valid data obtained}}{\text{total data needed}} \times 100$

12.5 Detection Limits

The sensitivity of an analytical method is related to the detection limit (i.e., the lowest concentration of an analyte that can be detected at a specific confidence level). Definitions of instrument detection limit (IDL), method detection limit (MDL), limit of quantitation (LOQ), and practical quantitation limit (PQL) follow in this section. The relationship of these terms is expressed graphically in figure 12-1.

- IDL - This is the smallest signal above background noise that an instrument can detect at a 99% confidence level. An IDL is measured by analyzing replicate blank samples. It is calculated by the mean plus two SDs for a normal distribution or three SDs for data which does not obey a normal distribution.

- MDL - This is the minimum signal level required to qualitatively identify a specific analyte by a specific procedure at a confidence level that is greater than 97%. An MDL is measured by analyzing a minimum of seven replicates spiked at 1-5 times the expected method detection limit. It is calculated by the SD times the student T-value at the desired confidence level.

- LOQ - This is the minimum signal level required to quantitate a specific analyte by a specific procedure at the desired confidence level (intralaboratory). An LOQ is measured by analyzing a minimum of seven replicates spiked at 1-5 times the expected MDL. It is calculated by 10 times the SD obtained in the MDL study.

- PQL - This is the minimum level that can be reliably achieved by a method within specified limits of precision and accuracy. A PQL is measured by the analysis of check samples containing analytes at concentrations of 1-5 times the MDL. The evaluation of interlaboratory check sample results is used to derive a PQL.

The MDL, LOQ, and PQL may be determined in a blank matrix or a specific sample matrix depending upon the objectives of the determination. Erco determines the MDL for routine methods using a blank matrix. MDLs are determined in a specific sample matrix when requested by the client as matrix-specific QA (see section 7).

12.6 Quality Assurance Objectives

Quality assurance objectives can be expressed in terms of precision, accuracy, and completeness. Table 12-1 lists data quality objectives for the project-specific parameters. Representativeness can be defined as the degree to which the data accurately represents the media from which it is collected. Representativeness can be measured by comparison of field duplicate results. Comparability expresses the confidence with which two data sets can be compared. Comparability can be measured by the adherence to QC practices and criteria contained in this plan.

Table 12-1. Quality assurance objectives[a]

Analyses	Matrix	Precision (% RPD)	Accuracy (% Recovery)	Completeness (%)
Volatile organics	Aqueous	<15	61-145	95
Volatile organics	Solid	<25	59-172	95
Semivolatile organics	Aqueous	<45	9-103	95
Semivolatile organics	Solid	<50	11-142	95
Pesticides, PCBs, and herbicides	Aqueous	<27	38-131	95
Pesticides, PCBs, and herbicides	Solid	<50	23-139	95
Metals, cyanide, and sulfide	Aqueous	<20	75-125	95
Metals, cyanide, and sulfide	Solid	<20	75-125	95
Dioxins and furans	Aqueous	<50	60-140	95
Dioxins and furans	Solid	<50	60-140	95

[a]The sources of these criteria are the EPA's SOW887 and "Data Quality Objectives for Remedial Response Activities Development Process," EPA/540/G-87/003.

13. CORRECTIVE ACTION

Corrective actions for laboratory problems are specified in Erco laboratory manuals and SOPs. Specific QC procedures are designed to help analysts determine the need for corrective action. Often, personal experience is most valuable in alerting the analyst to suspicious data or malfunctioning equipment. Corrective action taken at this point helps to avoid collection of poor quality data.

Problems not immediately detected during the course of analysis may require more formalized, long-term corrective action. The essential steps in the corrective action systems are as follow.

1. Identify and define the problem.
2. Assign responsibility for investigating the problem.
3. Investigate and determine the cause of the problem.
4. Determine a corrective action to eliminate the problem.
5. Assign and accept responsibility for implementing the corrective action.
6. Establish effectiveness of the corrective action and implement it.
7. Verify that the corrective action has eliminated the problem.

This scheme is generally accomplished through the use of the corrective action request form (figure 13-1) which is available to all Erco personnel. Using this form, any laboratory analyst or project member may notify the QA director of a problem. The QA director initiates the corrective action scheme by relating the problem to the appropriate laboratory managers and/or project managers who investigate or assign responsibility for investigating the problem and its cause. Once determined, an appropriate corrective action is approved by the QA director. Its implementation is later verified through a laboratory audit.

ENSECO Incorporated

ERCO

QUALITY ASSURANCE CORRECTIVE ACTION REQUEST

Originator: _____ Date: _____

Laboratory: _____ Project: _____

Problem: _____

Action Planned: Implemented:

QA Director: _____ Date: _____

Figure 13-1. Corrective Action Request Form.

Close scrutiny is paid to the quality and validity of the analytical data for any given analysis. Variations of more than two SDs (unless stricter controls are stipulated in the method used) will warrant corrective action procedures. The nature of such will be determined by the method employed. In most instances, a reanalysis (and possibly a recalibration) may be necessary to correct the problem.

Any and all corrective actions required for this project (either laboratory or field related) will be relayed to the Hatcher-Sayre project manager who will then be responsible for notifying the appropriate personnel and implementing project level corrective action such as resampling.

14. QUALITY ASSURANCE REPORTS TO MANAGEMENT

This reporting system is a valuable tool for measuring the overall effectiveness of the QA program. It serves as an instrument for evaluating the program design, identifying problems and trends, and planning for future needs. Divisional QA directors submit extensive monthly reports to the vice president of QA and the divisional director. These reports include the following items.

- The results of the monthly systems audits including any corrective actions taken.
- Performance evaluation scores and commentaries.
- Results of site visits and audits by regulatory agencies and clients.
- Performance on major contracts (including CLP).
- Problems encountered and corrective actions taken.
- Holding time violations.
- Comments and recommendations.

In addition, on a weekly basis, a summary of the 5% QA audit of reported data is sent to the corporate QA office.

The vice president of QA submits weekly reports to the chief executive office and monthly reports to the Enseco management committee and each divisional director. These reports summarize the information gathered through the laboratory reporting system and contain a thorough review and evaluation of laboratory operations throughout Enseco.

The most recent two to three months of analytical data are kept on-line. All other data are archived on magnetic tape or optical disk.

15.3 Laboratory Benchsheets

Laboratory benchsheets are used to document information from routine laboratory operations, including sample preparation and analysis. Benchsheets are used to ensure that the information is recorded in a complete and organized manner and that the analysis can be reconstructed if necessary. Portions of information from the benchsheet are also stored in the LDMS.

15.4 Laboratory Notebooks

Laboratory notebooks are used to document information that cannot easily be recorded in the LDMS. Information typically recorded in laboratory notebooks includes unusual observations or occurrences in the analysis of samples or method development information. Each page in a laboratory notebook is initialed and dated as information is entered.

15.5 Project Files

A project file is created for each project handled within the laboratory. The project file contains all documents associated with the project. This includes correspondence from the client, chain-of-custody records, raw data, copies of laboratory notebook entries pertaining to the project, and a copy of the final report. When a project is complete, all records are passed to the document custodian who inventories the file, checks it for completeness, and puts the file into document archive.

Freeman GW. Fld

State of Wisconsin

CORRESPONDENCE/MEMORANDUM

Date: February 13, 1990

File Code: 4430

To: Kathy Thompson - SW/3

From: Mark Tusler - SW/3

Subject: Freeman Chemical TAD Schedule

Freeman is currently on a December, March, June, and September TAD schedule. This sampling is being required under the authority of an EPA 3008(h) order. To facilitate lab scheduling, Freeman requested and EPA approved a January, April, July and October scheduling. Please revise the TAD dates to reflect this new schedule.

cc: Linda Freitag

HATCHER-SAYRE, INC.

February 2, 1990

Mr. William E. Muno, Chief
RCRA Enforcement Branch
USEPA, Region V
230 South Dearborn St.
Chicago, IL. 60604

Tusler

Re: 1989 Annual Report
Freeman Chemical Corp.
Saukville, Wisconsin
Job No. 0001-003

Dear Mr. Muno:

Attached is a copy of the 1989 Annual Report for the above referenced project. The report presents the data from the four (4) quarterly sampling periods conducted last year. Data from previous years were also included as appropriate.

The presentation of the data is in accordance with the agency approved project plan, except for the individual trend analyses and isoconcentration maps of benzene, toluene, ethylbenzene and xylene (BTEX). As discussed with Mr. Bob Smith in your branch, (January 29 and February 1, 1990) and Mr. Mark Tusler, WDNR (January 31, 1990), since these data mirror the data for total VOCs, the individual breakdown for each compound would not provide any additional useful information. What we provided instead was an analysis of four (4) additional compounds not included in the work plan. These compounds, trichloroethene, 1,2-dichloroethene (total), vinyl chloride and carbon disulfide, indicate possible off-site sources of contamination.

Based upon the data analysis, recommendations have been made concerning the parameters to be analyzed and the future sampling schedule. If these recommended changes are approved, a revised project work plan will be prepared and submitted to the agencies.

3150 Custer Drive, Suite 301, Lexington, Kentucky 40502 (606) 271-0269 Fax No. (606) 271-1204