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STAGE 1 & 2 WORKPLANS

DATE: Dec. 8, 1997

Laboratory Quality Assurance Project Plan

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ENVIRONMENTAL BUREAU OIL CONSERVATION DIVISION

Signature of the authorized individuals below constitutes approval of the general format and composition of this manual. Individual sections are coordinated with the parties responsible for their implementation.

Approved by:

12-12-97

Dr. Blair Leftwich, Laboratory Director

DATE

<u>2 Da 97</u> DATE

James Taylor, Laboratory Quality Assurance Officer

Document Control Number <u>154</u>

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1. Introduction

1.1 Preface

This Quality Assurance Plan is written in accordance with the elements required in *EPA Requirements for Quality Assurance Project Plans for Environmental Data Operations*, EPA QA/R-5, October 1997. This document contains the required elements of a Quality Assurance Plan and is prepared so that entire sections can be referenced in specific project plans.

The QA Plan defines the systems of quality control and quality assessment that make up the comprehensive Quality Assurance program at TraceAnalysis. Quality control consists of specific procedures applied to all phases of analysis from sample receipt through the final reporting of results. The purpose of quality control is to ensure that quality goals are met under routine operating procedures. Quality assurance involves the continuous evaluation of data and monitoring of analytical processes for the purpose of ensuring that the quality control systems are performing effectively.

1.2 Quality Policy

TraceAnalysis is committed to quality and expects from its management and staff a high degree of commitment to providing legally defensible data of known and appropriate quality to its clients. The validity and reliability of the data generated is ensured by the adherence to documented standard operating procedures SOPs TraceAnalysis' SOPs are written to comply with the laboratory management's interpretation of approved analytical methods.

TraceAnalysis emphasizes the application of sound quality assurance/quality control principles beginning with the initial planning of the project, throughout all analytical procedures, and ultimately with the preparation of the final report. The principles of the data quality objectives for representativeness, completeness, comparability, precision, and accuracy are applied to the analytical data generated.

To ensure client satisfaction, TraceAnalysis encourages strong interaction with the client at all phases of the project. Proactive interaction with the client helps TraceAnalysis to deliver a final product that meets the Quality Assurance Plan objectives as well as project-specific data quality objectives.

TraceAnalysis is committed to providing the resources, facilities, equipment, and personnel, to ensure the timely completion of analysis and adherence to applicable QA/QC protocols.

1.3 Fields of Testing Covered

TraceAnalysis provides environmental testing for industry, municipalities, regulatory agencies, and consulting and engineering firms using approved analytical methods. Analytical procedures fall into one of four categories of analysis: chemical analysis, physical analysis, trace metal analysis, and organic analysis. An extended list of testing methods is provided in Table 6-1.

1.4 Management of Quality Assurance Project Plan

1.4.1 Responsibility

The management of TraceAnalysis' Quality Assurance Project Plan is the responsibility of the Quality Assurance Officer.

1.4.2 QA Project Plan Review Frequency

The manual is reviewed yearly by the Quality Assurance Officer. At any time during the year, lab personnel may request changes to the manual based on actual lab practice. Request for revision of the manual must come directly to the quality assurance department and are reviewed for admission into the next quality plan revision.

1.4.3 Documentation of Revisions

The change description, revision date, and initials of the Quality Assurance Officer are recorded on the Revision Log located Appendix 1 of this manual. Each page of this manual contains the most recent revision number and revision date.

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2. Organization and Responsibility

2.1 Overview

TraceAnalysis operates a single laboratory located in Lubbock, Texas with sample pick-up personnel located in most major cities across the state. The laboratory occupies approximately 11,000 square feet of which approximately 8,500 square feet are dedicated to laboratory space. TraceAnalysis' floor plan is depicted in Figure 2-1.

Extraction and instrumentation labs are kept separate with individual HVAC units to minimize cross-contamination. Each area has a fire extinguisher and emergency notification device. Eye wash stations, safety shower and fire blanket are accessible to all areas where chemicals are used for sample extraction and preparation purposes. Safety storage cabinets are located in areas where flammable chemicals, acids, or corrosives are stored.

2.2 Roles and Responsibilities

2.2.1 Quality Assurance Officer

The Quality Assurance Officer (QAO) has the responsibility for ensuring that all activities of the lab are in compliance with quality policies. The QAO reports directly to the Director and has the authority and the responsibility to implement and approve corrective actions as needed. The QAO is responsible for monitoring QC sample analysis results and the results obtained for analyses of external performance samples to identify potential problems. He is responsible for initiating both preventive and corrective action processes as needed to ensure proper operations within the laboratory. The QAO is also responsible for maintaining certifications required for laboratory operations.

2.2.2 Laboratory Director

The laboratory is managed by a Laboratory Director with the responsibility for the technical and financial performance of the company. He also has the ultimate responsibility for all aspects of the Quality Assurance Plan. Other duties include client consultation, final data review, and signature of lab reports.

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Figure 2-1 Floor Plan



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2.2.3 Supervisory Personnel

An organics and inorganics section supervisor oversees technical operation in their respective areas. Responsibilities include sample scheduling, chemist training, instrument preventive maintenance, and raw data review.

2.2.4 Analysts and Technical Staff

Laboratory analysts and technicians are responsible for performing analyses according to standard operating procedures and for evaluating the acceptability of their data based on established quality control criteria. Analysts are also responsible for initiating corrective action when QC criteria are not met.

2.2.5 Marketing and Client Services

The department of marketing and client services provides the client interface and the project management staff necessary to ensure a project is complete in a manner required by the client.

2.2.6 LIMS Director

The LIMS Director is responsible for developing and maintaining the LIMS system including the networking of the laboratory. Computer training is also provided for the operation of the LIMS as well as other programs.

2.3 Organizational Structure

TraceAnalysis' organizational chart is depicted in Figure 2-2.

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Figure 2-2 Organizational Chart



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3. Quality Assurance Objectives

3.1 Introduction

The purpose of this Quality Assurance Plan is to define procedures for the establishment of analytical systems and for the acquisition, documentation, evaluation, reporting, and archival of legally defensible data of known quality. The objective is to provide uniform systems for sample receipt, sample handling, instrument maintenance and calibration, methods control, performance evaluation, data acquisition, evaluation of quality, and reporting of the data. Specific procedures to be used for maintaining chain of custody, sample receipt, storage and disposal of samples, preventive maintenance, chemical analyses, internal quality control, data reporting, QA audits and corrective actions are described in specific sections of this plan or in standard operating procedures SOPs which are referenced when applicable. This section addresses the objectives of accuracy, precision, completeness, representativeness, and comparability.

3.2 Precision and Accuracy

The QA objectives for precision and accuracy are to establish and maintain analytical systems that produce analysis results supported by QC data within acceptance criteria specified in the proposed analytical procedures. Precision and accuracy guidelines for the organic and inorganic procedures recommended by the USEPA are normally specified in the individual methods. These provide guidance as laboratory specific criteria are developed for each analytical method.

3.2.1 Organics

Due to the extensive number of organic compounds selected as target analytes and environmental sample matrices, the development of precision and accuracy objectives and control limits for each analyte in all potential matrices is impractical. Thus, information is obtained for water and solid matrices that are representative of the normal environmental sample types. This is accomplished by determining the percent recovery of (1) matrix spike and matrix spike duplicate compounds added to selected samples before extraction and analysis, (2) surrogate spike compounds which are added to every sample prior to extraction and analysis to test extraction efficiency and matrix effects (3) laboratory control samples (LCS), which are samples prepared from reference materials in DI water or clean sand and taken through the entire preparation and analysis procedure. Refer to Appendix 2.

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3.2.2 Inorganics

Precision and accuracy data for inorganic test parameters are obtained by analysis of duplicate samples or matrix spike and matrix spike duplicates as a measure of precision and matrix spike and laboratory control samples as a measure of accuracy. At least one duplicate sample or matrix spike sample is analyzed per sample matrix type per batch of samples or for each 10 samples analyzed, whichever is more frequent, or as specified by method requirements. The matrix spike recovery and relative percent difference (RPD) of the duplicates for each component is calculated for data assessment. Accuracy and precision data are obtained from LCS and LCS duplicate analyses when matrix spikes are not practical. Refer to Appendix 2.

3.3 Representativeness

Representativeness is a qualitative measure that is related to the ability to obtain a sample that best reflects the characteristics of the part of the environment that is to be assessed. The laboratory uses homogenization of the sample, if compatible with the tests to be performed, to ensure the results obtained are representative of the sample as received.

3.4 Completeness

Completeness is a measure of successfully obtaining all information necessary for a valid scientific study. The objective for completeness is: the methodology proposed for chemical characterization of the samples collected will provide data meeting QC acceptance criteria, following standard laboratory data review and validation, for at least 95% of all samples collected. Completeness may also be defined as a comparison of the number of tests successfully completed (with acceptable QC) to the number of tests requested. Nonconformance/corrective action (NC/CA) reports are completed in accordance with standardized procedures in order to provide explanation when QC criteria are not met. The NC/CA report is completed by the analyst describing the situation encountered. The corrective action required is taken and documented in the appropriate section of the NC/CA report by the supervisor and analyst. See Section 11, Corrective Action.

3.5 Comparability

Comparability is also considered during the preparation of the work plan. The objective of comparability is to produce results that do not differ significantly from those produced by other parties for the same purpose. TraceAnalysis uses SOPs based on EPA approved methods and procedures in order to achieve comparability with data from previous studies and from other laboratories. If an EPA approved

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procedure is not available or not required for the analyte(s) or matrix to be analyzed, alternative, published and/or validated procedures are followed. TraceAnalysis participates in external and intra-laboratory performance evaluation (PE) studies as an additional means of establishing comparability in the laboratory. In addition to participating in USEPA Water Pollution (WP) studies, and analyzing PE samples for various government agencies and commercial clients, TraceAnalysis also participates in intra-laboratory double-blind studies.

4. Sample Handling Procedures

4.1 Sample Containers

TraceAnalysis provides its clients with precleaned sample containers necessary to carry out any given project. Sample containers are constructed of polyethylene or glass, as listed on Tables 4-1A and 4-1B. TraceAnalysis routinely uses new sample containers obtained from a supplier that has been qualified, by the laboratory's analysis of DI water blanks to deliver containers free from contamination. Sample containers are used only once and disposed of according to federal, state, and local guidelines. In addition, TraceAnalysis can supply certified, precleaned containers upon request

4.2 Sample Container Orders

4.2.1 Bottle Order Form

Bottle orders are prepared by the Support Services Manager using a bottle order form or equivalent (Figure 4-1) Bottle orders must be placed as far in advance as possible. The bottle order form must contain the following information: analyses, matrices, number of samples, name and shipping address, and date required.

4.2.2 Trip Blanks

Trip blanks are prepared by completely filling a 40 mL VOA vial with organicfree reagent water. The last few drops are gently poured into the vial so that surface tension holds the water in a convex meniscus. The vial is then capped. If air space is present, the procedure is repeated. The vial is labeled as a trip blank. Preparation of field blanks is dependent on their function, i.e. preserved or nonpreserved.

4.2.3 Filling Bottle Orders

The bottle order is filled by a member of the support services group. Bottles, with proper preservatives added, are packed in shipping containers using bubble-wrap to minimize breakage. The containers are typically sent in the ice chest in which they will be returned to the lab. A chain-of-custody form is sealed in a plastic bag and sent with each bottle order. Labels and custody seals are also made available. The bottles may be picked up at the laboratory, delivered by TraceAnalysis personnel or a third party carrier.

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4.3 Definition of Holding Times and Preservatives

Holding times are defined as the amount of time that elapses between the collection of the sample from the source in the field and the beginning of the analysis procedure. Preservatives are defined as techniques used to maintain the target analytes at concentration representative of those in the source sampled until the sample is analyzed in the laboratory. Published holding time is viewed as valid as long as the associated preservation and container requirements have been met. Table 4-1 lists appropriate sample containers, preservatives, and maximum holding times for each analysis.

4.4 Definition of Turn Around Time

Turn-around-times are defined as the amount of time that elapses between the receipt of the sample at the lab and the receipt of the data by the client, excluding weekends and holidays. Reports are faxed upon completion with hard-copy final reports mailed thereafter.

4.5 Chain-Of-Custody

A chain-of-custody record (Figure 4-2 or equivalent) should be completed with each sampling event to document sample custody from the time of collection through transfer of custody to the laboratory. At a minimum, the chain-of-custody record must contain the following information: analyses required, type of sample bottle, preservative, sample identification, signature of collector, date and time of collection, and signature and inclusive date and times of possession for each person taking custody of the samples.

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Figure 4-1 Bottle Order Form

TraceAnalysis, Inc. Sample Bottle Order Form

Sample kits and regular shipping costs provided at no charge. Please allow 5 days for shipping. For overnight shipping, please include your carrier account number. All sample kits include: chain of custody, labels, custody seals, return airbill, permanent marker and plastic bags for ice.

All coolers received must be 4° C and will be noted on chain-of-custody.

Date Ordered	Date Needed	
Send to:	Location:	······
Attention:		
Phone #:	Project #:	

Analysis	Number of	Samples	Analysis	Number of Samples		
	Solid	H2O		Solid	H20	
BTEX			VOCs			
трн		-	SVOCs			
РАН			Metals			
TDS			TCLP (Metals, Vol, SV)			
Lead			RCI			
Other						

Special	Instructions:
• p • • • •	

: Yes No
Packed by:

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PIOH CHAIN-OF-CUSTODY RECORD AND ANALYSIS REQUEST bisbrists moil frieisith li emit bruoiA riut H9 ,821 ,008 ANALYSIS REQUEST (Circle or Specily Method No.) Pest. 608/608 PCB's 8080/608 GC/MS Semi. Vol. 8270/625 **REMARKS**: CC.MS Vol. 8240/8260/624 2 IJЯ LAB Order ID # TCLP Semi Volatiles TCLP Volatiles Y / N LAB USE ONLY TCLP Metals Ag As Ba Cd Cr Pb Hg Se λ / N Total Metals Ag As Ba Cd Cr Pb Hg Se Log-in Review 0758 HA9 Headspace нат Intact Temp 111BE 8020/602 BTEX 8020/602 6701 Aherdeen Avenue, Ste. 9 Lubhock, Texas 79424 зміт SAMPLING Tel (806) 794 1296 Fax (506) 794 1298 этаа PRESERVATIVE METHOD Timo: Time: Time: 1 (800) 378 1296 NONE ICE Submitted of samples constitutes agreement to Terms and Conditions listert on reverse side of C.O.C. CONH Sampler Signature: Date: Dato: Date: тон Project Name: Phone #: SUDDEE Received at Laboratory by: MATRIX Fax #: ЯIA SOIL RETAW Received by: Received by: JnuomA\amuloV TraceAnalysis, Inc. * CONTAINERS Tinte: Time: Time: FIELD CODE Date: Date: Date: Iclinquished by: Relinquished by: Relinquished by: Project Location: ameN ynedmo ontact Person: (LAB USE) Project #: ivoice to: -ddress:

Figure 4-2 Chain-of-Custody

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Table 4-1ASample Preservative Requirements

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PARAMETERS	Container	Preservativo	Maximum	Sample
			Holding Time	Volume
INORGANICS/PHYSICALS	EN SU CONTRACTOR		•	
Alkalinity	P,G	Cool, 4°C	14 Days	250 mls
Ammonia	P,G	Cool, 4"C, H.SO to pH<2	28 Days	250 mls
BOD	P,G	Cool, 4"C	48 Hours	500 mls
COD	P,G	Cool, 4"C, H SO to pH<2	28 Days	100 mls
· CEC	P,G	HNO to pH<2	6 Months	250 mls
Chloride	P,G	None	28 Days	250 mls
Chlorine, Residual	P,G	None	Analyze Immediately	100 mls
Chromium, Hexavalent	P,G	Cool, +"C	24 Hours	250 mts-
Conductivity	P,G	Cool, 4"C	28 Days	100 mls
Cyanide, Amenable	P,G	Cool, 4°C, NaOH to pH>12	14 Days	500 mls
Cýanide, Total	P,G	Cool, 4"C, NaOH to pH>12	14 Days	500 mls
ESP	P,G	HNO to pH<2	6-Months	250 mls
Flashpoint	P,C 1	None	14 Days	100 mls
Fluoride	P	Cool, 4"C	28 Days	100 mls
Formaldehyde	P,G	Cool, 4"C	NA	250 mls
Hardness	P,G	HNO, to pH<2	6 Month's	250 mls
Nitrale	P,G	Cool, 4°C	48 Hours	100 mls
рН	P,G	None	Analyze Immediately	50 mls
Phosphate	P,G	Cool, 4°C	48 Hours	100 mls
Phosphorous, Total	P,G	Cool, 4"C, H SO to pH<2	28 Days	250 mls
SAR	P,G	HNO, to pH<2	6 Months	250 mls
Specific Gravity	P,G .	None	NA .	100 mls
Sulfate	P,G	Cool, 4"C	28 Days	100 mls
Sulfide	P,G	Cool, 4"C, Zinc	7 Days	100 mls
	1 . 1	Acetate+NaOH to pH>9		
Total Kjeldahl Nitrogen	P,G	Cool, 4"C, H,SO to pH<2	28 Days	500 mls
Total Dissolved Solids	P,C	Cool, 4"C	7 Days	250 mls
Total Solids.	P,G	Cool, 4°C	7 Days	250 mls
Total Suspended Solids	P,G	Cool, 4"C	7 Days	250 mls
Total Volatile Solids	P,G	Cool, 4°C	7 Days	250 mls
METALS	· · · · · · · · · · · · · · · · · · ·	2	and the second second second	
Metals except Boron,	P,G	HNO, to pH<2	6 Months	250 mls
CR6+, and Mercury	ا _ن ا			
Mercury	P,G	HNO, to pH<2	28 Days	250 mls -
Boron	P 1	HNO, to pH<2	6 Months	250 mls
ORGANICS	10 10 10 10 10 10 10 10 10 10 10 10 10 1			
Aromatic Volatiles (BTEX)	G W/TLS	Cool, 4"C, HCL to pH<2	14 Days	2 VOAs
Oil and Grease	G W/TLC	Cool, 4°C, HCL to pH<2	28 Days	500 mls
Organo-Chlorine Pesticides	G W/TLC		7 Days	1 L
Phenols	G W/TLC	Cool, 4"C	28 Days	- 1 L
Polynuclear Aromatic Hydrocarbons	G W/TLC	Cool, 4"C.	7 Days	1L
Semi-Volatile Organics by CC/MS	G W/TLC	Cool, 4"C	7 Days	2 L
Total Organic Carbon (TOC)		Cool, 4°C, H SO to pH<2	28 Days	2 VOAs
Total Organic Halides (TOY)	- <u>C</u>	Cool, 4"C. H SO	7 Days	500 mls
Volatile Organics by CC/MS	- <u>-</u> G/TIS-	Cool, 4"C, HCL to pHc?	14 Days	2 VOAs
TRPHC	G w/TIC	Cuol. 4"C. HCL to pH<2	14 Days	500 mls
	G W/TIS	Cuol 4"C HCL to oH<2	14 Days	2\'()As
TPH CRO	- G W/TIS	Coul 4"C HCL to pH<2	14 Days	2 \'OAs
Find Finnensint	- <u>C/TIC</u>	Cool 4"C	14 Days	2 VOAs
RCI	<u>PC</u>	Cool 4"C	14 Days	500 mlc
KCI	1,0	C001, 4 C	19 0433	200 mis

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Table 4-1BSample Preservative Requirements

PARAMETERS	Container	Preservative	Maximum Holding Time	Sample Volume
VOLATILE ORGANICS	an et en same			
Concentrated Waste	G w/TLC	Cool, 4°C	14 Days	4 oz.
Soil/Sediment	G w/TLC	Cool, 4°C	14 Days	4 oz.
Sludge	'G w/TLC	Cool, 4"C	14 Days	4 oz.
SEMI-VOLATILE ORGANICS				
Concentrated Waste	G w/TLC	Cool, 4"C	14 Days until extraction, 40 Days thereafter	4 oz.
Soil/Sediment	G w/TLC	Cool, 4"C	14 Days until extraction, 40 Days thereafter	4 oz.
Sludge	G w/TLC	Cool, 4°C	14 Days until extraction, 40 Days thereafter	4 oz.
PCB (in Transformer oil)	P,G.	Cool, 4"C	28 Days, recommended	20 mls

- NOTES:
- Abbreviations:
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- G: glass P: polyethylene
- TLC: Teflon-lined cap
- TLS: Teflon-lined septum
- Sample Preservation:

Sample preservation should be performed immediately upon sample collection. For composite samples, samples may be preserved by maintaining the sample at 4°C until compositing and sample splitting is completed.

• Holding Times:

Holding times listed are the times that samples may be held before analysis and still be considered valid under EPA regulations. Holding times are measured from the date of sampling.

Measurements:

If the dissolved content is to be measured, samples should be filtered on-site immediately before adding preservatives.

These requirements are based on 40CFR136, Table II; SW-846, third edition, revision 1.

PLEASE CONTACT THE LABORATORY FOR SAMPLE VOLUMES REGARDING MULTIPLE ANALYSES.

5. Sample Custody and Handling

5.1 Chain-of-Custody

All samples received by TraceAnalysis are accompanied by a chain-of-custody form completed by the client and/or sampler. TraceAnalysis provides chain-of-custody forms for all containers supplied to the client. Clients choosing to use chain-ofcustody forms other than those provided by TraceAnalysis are responsible for ensuring all essential information is included on the form. Figure 4-2 shows the TraceAnalysis chain-of-custody form.

5.2 Sample Receiving

5.2.1 Sample Receipt and Verification of Documentation

Samples are received in accordance with the procedures set for the in TraceAnalysis sample receipt and log-in SOP. Shipping containers are inspected for custody seals and the condition is noted in the bottom right hand corner of the chain-of-custody. The shipping containers (usually ice chests) are then opened and inspected for enclosed documentation. The sample bottle labels are inspected and compared to the chain-of-custody. Any discrepancies are noted on a nonconformance/corrective action (NC/CA) report form and the client is notified by client services personnel to determine the corrective action required. Samples received with no paperwork are held in refrigerated storage until the proper instruction for analysis is received. The client is contacted immediately and the resolution of the problem is documented in the project file.

5.2.2 Verification of Sample Preservation

The temperature inside the shipping container is determined using a hand-held infrared thermometer and recorded on the chain-of-custody form. The pH of preserved aqueous samples is verified as soon as possible after sample receipt and recorded on the internal chain-of-custody form. The pH of preserved VOA vials is measured at the time of sample preparation and recorded in the instrument run log.

5.2.3 Rejection of Samples

If samples are received improperly preserved, broken, or with headspace in VOA vials, the client is notified to determine corrective action. This is recorded on a NC/CA report form.

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5.3 Sample Log-In

Samples are logged into TraceAnalysis Laboratory Information Management System (LIMS) in accordance with the sample receipt and log-in SOP. Each project is assigned a unique batch code and each sample receives a unique sample number. Upon entry of all required sample identification and analysis information into the LIMS, the information is reviewed by the laboratory director for accuracy and completeness. Any errors or omissions are corrected at this time.

5.4 Sample Storage

The samples are stored in designated refrigerators according to the type of analysis to be performed. Samples to be analyzed for volatile organic compounds are stored in separate refrigerators designated for volatile samples (VOA vials only). All refrigerators are monitored daily to ensure the temperature is maintained within a range of 2-5° C. Deviations from this temperature range are noted in the temperature logbook and corrective action is taken immediately to ensure the integrity of the samples.

5.5 Sample Access and Internal Chain-of-Custody

Integrity of samples is ensured by operating the laboratory facilities under controlled access. Only employees are allowed into the laboratory areas. Visitors must register upon arrival and allowed access to the facility only with an escort. An internal chain-of-custody for samples, is also maintained within the laboratory. Analysts must check the samples in and out of cold storage on the provided internal chain-of-custody form.

5.6 Confidentiality

TraceAnalysis is responsible for seeing that confidentiality is maintained by its employees concerning all confidential information with which they become acquainted as a result of their contact with any given project. Trace agrees to hold all disclosed confidential or proprietary information or trade secrets in trust and confidence. The information shall be used only for the clients purposes. The information shall not be disclosed to any third party without written consent of the client.

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5.7 Sample Subcontracting Documentation

When samples are subcontracted to other laboratories due to TraceAnalysis' inability to perform the required analyses, a separate chain-of-custody record and analysis request is filled out and sent accompanying the samples. A copy is also put into the client file. Subcontracted work is reported on the letterhead of the laboratory which performed the analysis. The client will be notified in advance of the subcontracted work. TraceAnalysis will have on file the QAPP and any other QA records needed of the subcontracting laboratory.

5.8 Sample Disposal

Samples not totally consumed during analysis and any excess extracts or digests generated during analysis are disposed of in accordance with local, state, and federal regulations. Specific disposal requirements are arranged with the client before receipt of samples. Sample disposal is addressed in the sample disposal SOP.

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6. Analytical Procedures

6.1 Purpose and Applicability

This section specifies the sources of analytical methods used by TraceAnalysis and defines controls on standard operating procedures SOPs, their content, approval for use, distribution, and revision. A list of preparation and analytical methods performed at TraceAnalysis is included as Table 6-1.

6.2 Laboratory Reference Documents

TraceAnalysis employs analytical methods from the following recognized sources:

40 CFR Part 136, Code of Federal Regulations,

EPA-600/4-79-20, Methods for Chemical Analysis of Water and Wastes, March 1983 Revision and updates

SW-846, <u>Test Methods for Evaluating Solid Wastes</u>, November 1986, Third Edition and updates

EPA/600/4-91/010, Methods for the Determination of Metals in Environmental Samples, June 1991

Methods of Soil Analysis, American Society of Agronomy, 2nd Edition, 1982

American Society for Testing of Materials

Standard Methods for the Examination of Water and Wastewater, 17th Edition, 1987

6.3 Method Modifications

Method modifications are permissible to allow for advances in analytical techniques as long as the actual chemistry of the method is not changed. An example of this would be modifying a gas chromatographic method for the use of capillary columns. Other permissible modifications would include sample volume or weight changes as long as the ratios set forth in the methods are retained.

6.4 Standard Operating Procedures

All analytical procedures are performed according to a written standard operating procedure, incorporating specifics regarding TraceAnalysis' quality control procedures, set-up, and operation of current laboratory instrumentation. SOPs address the following:

- <u>Method Reference</u>-Lists the reference document from which the SOP was derived.
- <u>Scope and Application</u>-Lists the property, analyte(s), or class of compounds measured by the method and summarizes the procedure. Describes the sample matrices working ranges, and situations to which the procedure applies.
- <u>Equipment</u>-Describes the instruments, glassware, and other equipment _ applicable to the procedure.
- <u>Reagents</u>-Describes the reagent and standard concentration grade, preparation, and use.
- <u>Procedure-Describes the sequence of activities to be performed</u>. This includes calibration or standardization, sample pretreatment, sample analysis, calculations, reporting limits, quality control checks, and special glassware cleaning procedures as appropriate to the method.
- <u>Corrective Action</u>-Describes corrective actions taken when nonconformance or out-of-control situations occur during the procedure. This includes invalid calibrations, method blanks, quality control checks, and matrix interference.
- <u>Preventive Maintenance</u>-Describes daily or routine maintenance procedures on instrumentation or equipment used in the analytical procedure.

All SOPs are approved by the appropriate Section Supervisor, the Quality Assurance Officer and Laboratory Director. Master copies of SOPs are retained by the Quality Assurance Officer. SOP documentation will be controlled by the use of a master log indicating the document, the identity of the recipient, the issue date, and the date collected from circulation. Any obsolete copies that remain in circulation will be marked as such. Controlled copies are distributed to appropriate areas of the laboratory. SOP revisions must be approved and distributed in the same manner as the original method.

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6.5 Method Start-Up QC

Before a method is approved to generate client data, method start-up quality control must be performed by each analyst and retained on file. Method start-up QC is method dependent and includes at a minimum: Initial Demonstration of Capability and Method Detection Limit Studies. If a modification is made to a method, the method start-up QC must be repeated with the modification as an integral part of the method. Method detection limit studies must be conducted at a minimum of once a year.

6.5.1 Initial Demonstration of Capability

The laboratory must demonstrate that it can meet the specifications in the method for recovery of analytes spiked into a reference matrix (reagent water). The test consists of spiking the analytes of interest into a set of four portions of reagent water and processing these aliquots through the entire analytical procedure. The mean concentration and the standard deviation of the mean concentration are calculated for each analyte, and these values are compared to specifications in each method. If the mean and standard deviation are within limits, the laboratory can use the method to analyze client samples.

6.5.2 Method Detection Limit Studies

The procedure for determining MDLs is published in 40 CFR part 136, Appendix B. All programs across the EPA cite this reference. The study is based on repetitive analysis of an interference-free sample spiked with a known amount of the target analyte. In general, seven aliquots of the spiked sample are taken through the entire sample preparation and instrument analysis protocol. The standard deviation of the results is determined, then multiplied by the one-tailed t-statistic at the 99% confidence level for the number of degrees of freedom in the study (one less than the number of repetitions used to calculate the standard deviation). For seven repetitions, this is a multiplier of 3.143. The resulting value is finally compared to the spike level in the sample. If the spike level is within five times the calculated MDL then the procedure is considered successful. If not, the spike level in the sample is adjusted, and the study is repeated.

6.6 Method Selection

TraceAnalysis will use EPA approved methodology for the analysis of environmental samples whenever such methods are available. If the client has not specified an approved method, TraceAnalysis will select a recognized and validated method for use.

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6.7 Equipment

An extensive scope of services, which TraceAnalysis provides to its clients, requires significant capital investment. The laboratory furnishes all items of equipment required for the correct performance of tests.

6.7.1 List of Equipment

A list of all major equipment is included in Appendix 5. It is not intended to be a compilation of all equipment in use, but simply a summary of the significant instrumentation dedicated to providing clients with on-time, quality data.

6.7.2 Maintenance

Daily and preventive type maintenance measures are recorded in logs located by the major pieces of instrumentation. Entries include source cleanings, injector maintenance, torch replacement, etc. Major repairs are also recorded and the field service technician's report is retained on file. Any item of equipment which has been subjected to overloading or mishandling, or which gives suspect results, or has been shown by verification or otherwise to be defective, shall be taken out of service, clearly identified and wherever possible stored at a specified place until it has been repaired and shown by calibration, verification or test to perform satisfactorily. The laboratory shall examine the effect of this defect on previous tests and notify the customer of any discrepancies in their reports.

6.8 Reagents, Solvents, Gases, and Outside Supplies

6.8.1 Reagents

All reagents are prepared using, at a minimum, ACS reagent grade chemicals. Preparation information is recorded in the reagent log in the wet chemistry lab.

6.8.2 Solvents and Acids

Solvents and acids used are ultra high purity typically surpassing those recommended in actual analytical methods. For volatile organics, purge and trap grade methanol is used. For extractable organics, pesticide grade or Optima TM grade solvents are used. For metals, trace metals grade acids are used.

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6.8.3 Gases

Carrier gases (He) used on gas chromatographs is UHP grade except for the GC/MS systems which use a research grade of carrier gas. Make-up and other gases used on gas chromatographs are selected according to manufacturer's specifications.

6.8.4 Outside Supplies

Although rare, if the laboratory procures outside supplies, they will not be used until they are inspected and verified as compliant to standard specifications relevant to tests concerned.

6.9 Glassware Specifications

All laboratory volumetric glassware (pipets and flasks) is Class A. When preparing standards and reagents, Class A volumetric glassware must be used.

6.10 Glassware Cleaning

All dirty laboratory glassware is cleaned and processed by the Support Services _ department. EPA approved cleaning and baking procedures are followed. Details of glassware cleaning are addressed in the glassware processing SOP.

6.11 Reagent and Reference Material Storage

6.11.1 Reagent Storage

All reagents and chemicals are stored in appropriate areas of the laboratory where they are most commonly used. Corrosive and flammable chemicals are stored in approved cabinets. Ionic salts and other dry chemicals are stored on shelves in the wet chemistry lab.

6.11.2 Reference Material Storage

Standards and reference materials are stored in the areas of the laboratory where used by analysts. A standards refrigerator or freezer designated for its use is in each area. Temperature logs are maintained for standards refrigerators and freezers.

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Table 6-1 Methods

Method Number

Parameter Inorganics Alkalinity Ammonia **Biological Oxygen Demand** Bromide Bulk Density Chemical Oxygen Demand Cation Exchange Capacity Chloride Chromium, Hexavalent Conductivity Corrosivity Cyanide Flashpoint/Ignitability Fluoride Formaldehyde Hardness Moisture Content Nitrate Nitrite рH Phenolics Phosphate Phosphorous Reactivity Specific Gravity Sulfate Sulfide Total Kjeldahl Nitrogen Total Dissolved Solids **Total Solids** Total Suspended Solids Total Volatile Solids

Organics Aromatic Volatiles BTEX Fuel Fingerprint Fractional Organic Carbon Ignitability 310.1 350.2 405.1 300.0 30-2 410.4 9081 300.0, 9056 7196A, 3500-Cr 120.1, 9050A 1110 335.2, 9010A 1010/7.1.2.1-4 340.2, 300.0, 9056 NIOSH 3500 SM 2340-B **ASTM D2216** 300.0, 9056 300.0, 9056 150.1, 9040B, 9045C 420.1 365.2 200.7 7.3.3.2/9010A, 7.3.4.2/9030A ASTM D 854-92 300.0, 9056 376.1 351.3 160.1, SM 2540 C 160.3, SM 2540 B 160.2, SM 2540 D 160.4, SM 2540 E

8021B/602 8021B/602 8015B 2974 7.1.2.1-4

[•]Modified from Methods for Soil Analysis, C.A. Black.

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Parameter

Method Number

413.1, 9070
8081A/608
80812/608
8270C/625
8270C/625
8270C/625
415.1,9060
9020B
8260/624

Metals

Aluminum, Al Antimony, Sb Arsenic, As Barium, Ba Beryllium, Be Boron, B Cadmium, Cd Calcium, Ca Chromium, Cr Cobalt, Co Copper, Cu Iron, Fe Lead, Pb Magnesium, Mg Manganese, Mn Molybdenum, Mo Nickel, Ni Potassium, K Selenium, Se Silicon, Si Silver, Ag Sodium, Na Strontium, Sr Thallium, Tl Tin, Sn Vanadium, V Zinc, Zn

6010B/200.7 6010B/200.7/7041/204.2 6010B/200.7/7060A/206.2 6010B/200.7 6010B/200.7 6010B/200.7 6010B/200.7/7131A/213.2 6010B/200.7 6010B/200.7/7191/218.2 6010B/200.7/7201/219.2 6010B/200.7/7211/220.2 6010B/200.7 6010B/200.7/7421/239.2 6010B/200.7 6010B/200.7 6010B/200.7 6010B/200.7/7521/249.2 6010B/200.7 6010B/200.7/7740/270.2 6010B/200.7 6010B/200.7/7761/272.2 6010B/200.7 6010B/200.7 6010B/200.7/7841/279.2 6010B/200.7 6010B/200.7 6010B/200.7

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7. Calibration Procedures

7.1 Introduction

Analytical instruments and equipment used to obtain measurements or record data to be used for calculations of analytical results are calibrated at a frequency and in a manner such that accuracy and reproducibility are consistent with the manufacturer's specifications for proper instrument operation, and the calibration is in compliance with the analysis method requirements.

Laboratory measurements are based upon comparisons to results obtained for the analysis of reference standards analyzed by the same method. The results obtained for the analysis of calibration standards are used to prepare calibration curves or calculate calibration factors. The results of the sample analysis are quantified using either internal or external calibration techniques. Typically, calibration is achieved by the analysis of five calibration standards for organics and three calibration standards for inorganics at concentration levels set forth in the referenced method.

7.2 Calibration Frequency

Recalibration of instrumentation is performed at specified time intervals, when indicated by the continuous verification procedures, or when required by the contractrequired method. Calibration procedures are method specific. Consult the appropriate SOP for details regarding initial and continuous calibration.

7.3 Secondary Reference

Where applicable, another source standard from an alternate accredited vendor is used to check the calibration of the instruments.

7.4 Calibration Records

Each analyst maintains calibration files for the methods performed. This file includes date of calibration, calibration plots, and calibration raw data. Data is also present showing traceability to standard reference materials.

7.5 Traceability of Calibration Reference Materials

All instruments are calibrated using standard solutions of known concentrations. The standards are prepared from certified reference materials traceable to NIST or from reference materials whose concentration has been verified against NIST traceable

materials. Certificates of Analysis from standards vendors are kept on file by the Quality Assurance Officer. Thermometers and balances are calibrated annually, or sooner as needed, using NIST traceable thermometers and weights. Daily verification of balance calibration is described in the balance calibration verification SOP. Balances, NIST weights and thermometers are serviced by certified technicians annually.

7.6 Standards Preparation Records

Calibration standards are prepared from commercially available traceable stock standard solutions. The identity of the stock solution, intermediate solution, preparation procedure, solvent lot, date, preparer, expiration date, and identity of the calibration standard are recorded in a standards preparation logbook. The entry is dated and signed by the analyst.

7.7 Acceptance of a Standard Curve

The laboratory calibration procedure utilized must meet or exceed the method calibration criteria for analyses performed. In the event that calibration criterion are not attained, a recalibration is attempted. If the calibration fails again, the analysis is halted and corrective action is taken. The calibration procedure in the method is followed for each specific analysis. Calibration procedures are documented on computer printouts, in the analysis logbook, and/or on bench sheets where applicable.

7.8 Initial Calibration Verification

The initial calibration verification (ICV) is performed to determine if the calibration curve that has just been generated is valid. It is a check upon the accuracy of the individual calibration standards used to perform the calibration. The ICV solution is prepared form a different lot number or obtained from a different manufacturer than that of the calibration standards.

7.9 Continuing Calibration Verification

The continuing calibration verification (CCV) is used to ascertain that the initial calibration is still holding and correct as the instrument is used to process samples. For instruments that incorporate analyte identification into the procedure such as retention time matching from a gas chromatograph or spectral matching from a mass spectrometer, the CCV also serves to determine that the identification criteria are still being met. The source of the CCV could be one of the calibration standards.

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8. Preventive Maintenance

8.1 Facility and Safety Requirements

Extraction and instrumentation areas are kept separate with individual HVAC units to minimize cross-contamination. Each area has a fire extinguisher and emergency notification device. Eye wash stations, safety shower, and a fire blanket are accessible to all areas. Ventilation hoods are located in all areas where chemicals are used for sample extraction and preparation purposes. Safety storage cabinets are located in area where flammable chemicals, acids, or corrosives are stored.

8.2 Instrument Preventive Maintenance

All instruments and equipment receive routine preventive maintenance, which is recorded in instrument specific maintenance logs. Routine maintenance ensures that the equipment is operating under optimum conditions, reducing the possibility of instrument malfunction.

Preventative maintenance procedures including lubrication, source cleaning, detector cleaning, and the frequency of such maintenance are performed according to the procedures recommended in the manufacturer's instrument user manual. Chromatographic carrier gas purification traps, injector liners, and injector septa are replaced on a regular basis. Precision and accuracy data are examined for trends and excursions beyond control limits to determine evidence of instrument malfunction. Maintenance must be performed when the instrument begins to degrade as evidenced by the degradation of peak resolution, shift in calibration curves, decreased sensitivity, or failure to meet one or another of the quality control criteria.

Instrument logbooks containing maintenance and repair records are kept in the laboratories at all times. The laboratories also maintain adequate supplies of spare parts such as GC columns, syringes, septa, injection port liners, and other consumable parts to minimize potential instrument downtime. In the event of equipment malfunction that cannot be readily resolved by laboratory personnel, service is obtained from the instrument vendor or manufacturer.

8.3 Contingency Plan

In the event of instrument failure, every effort will be made to analyze samples within holding times by approved alternate means. If duplication of instrumentation is
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insufficient to handle the samples of concern, efforts will be made to secure the equivalent or similar analyses from another approved laboratory. The client will be consulted immediately in the eventuality.

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9. Laboratory Quality Control

9.1 Purpose and Applicability

This procedure provides an overview of the quality control (QC) measures used to assess and control analytical processes at TraceAnalysis. Specific information on quality control checks for individual laboratory departments is provided in TraceAnalysis SOPs for individual analysis methods.

9.2 Responsibilities

9.2.1 Quality Assurance Department

The Quality Assurance Department shall establish and publish acceptance limits for quality control checks and assist laboratory personnel in updating variable limits annually, at a minimum.

9.2.2 Laboratory Analysts

Laboratory analysts shall compare the results of quality control checks to the published acceptance limits, and shall take appropriate corrective measures whenever acceptance limits are exceeded. Corrective measures shall be documented.

9.3 Daily Quality Control

NOTE: The following discussion of the daily quality control program is general in nature. The test specific requirements of the methods, as outlined in TraceAnalysis SOPs, supercede these general requirements. In addition, client or project-specific QC requirements may supercede those specified in this QA Plan.

9.3.1 Quality Control Checks

The daily quality control program includes a variety of QC checks inserted in the analysis process by analysts. These checks include instrument tuning or sensitivity checks, continuing calibration or calibration verification standard, and lab control sample results are calculated as percent recovery. Method blank results are evaluated for the presence/absence of laboratory contaminants. These quality control checks monitor the accuracy of the analytical procedure in the absence of matrix interference. The decision to accept or reject analytical

procedure in the absence of matrix interferences. The decision to accept or reject analytical results is based on these quality control results.

9.3.2 Acceptance/Rejection Limits for QC

Acceptance limits for these checks are taken from EPA methods or are established by TraceAnalysis from actual QC data. If these checks fail to meet acceptance limits, corrective action is required before continuation of analysis and/or reporting of the data. The corrective action taken for each out-of-control event must be described in the analysis log and approved by the data reviewer. In addition, a nonconformance/ corrective action form is completed and kept with the raw data.

9.3.3 Matrix Spikes

One in ten (one in twenty samples for GC analysis) of a similar matrix is analyzed in duplicate and as a matrix spike, or as a duplicate matrix spikes, to evaluate matrix effects on analyte recovery. Accuracy is calculated as percent recovery of the matrix spike. Precision is calculated as the relative percent difference (RPD) of duplicates or matrix spike duplicates.

9.3.4 Acceptance/Rejection Limits for MS/MSD

Acceptance limits for these checks are also taken from EPA methods or are established internally by TraceAnalysis. If matrix spike recovery fails to meet acceptance limits and the analytical system yielded acceptable results for calibration standards, lab control samples, and surrogate standards, the sample is re-extracted and analyzed again. If matrix spike recovery fails to meet acceptance limits again, the associated sample results are qualified to indicate the probable presence of matrix interference. If precision acceptance criteria are exceeded, reanalysis of the duplicates and all of the positive samples in the batch is required. The corrective action taken is described in the analysis log and approved by the data reviewer. In addition, a nonconformance/corrective action form is completed and kept with the raw data.

9.4 Acceptance Limits

Acceptance limits for the daily QC program are taken from EPA methods or are established by TraceAnalysis from actual data as described in this section. Acceptance limits are calculated and summarized annually, at a minimum, and distributed to laboratory operations personnel by the Quality Assurance Officer. Current QC acceptance limits are listed in the Appendix #3 of this QA Plan.

9.4.1 Fixed Limits

In general, acceptance limits for GC, GC/MS and metals analyses for tuning, initial and continuing calibration, method blanks, and precision and accuracy of matrix spikes and duplicates or duplicate matrix spikes are based on acceptance limits established in EPA methods.

9.4.2 Variable Limits

Variable limits are based on laboratory-generated data and are updated annually, at a minimum.

Acceptance limits for percent recovery of lab control samples and GC and GC/MS surrogate standards are calculated from actual QC data. The mean (x) and standard deviation (s) are calculated from the most recently generated percent recovery data. A minimum of 20 values is necessary to establish limits. Outliers are excluded from the calculation of acceptance limits. Control charts are available for these parameters.

Acceptance limits are calculated as follows, where (x) represents the individual values and (n) is the number of values.

Parameter	Symbol	Formula
Upper Control	UCL	x + 3s
Upper Warning Limit	UWL	x + 2s
Center Line (mean)	x	(Σχi)/n
Lower Warning Limit	LWL	x – 2s
Lower Control Limit	LCL	x – 3s

If the data generated are insufficient to calculate acceptance limits and the method does not provide acceptance criteria, the following limits will apply:

- Inorganic Chemistry: 75-125% recovery
- Metals: 75-125% recovery (water); 50-150% recovery (soil)

- Organic chemistry-Volatiles: 75-125% recovery
- Organic chemistry Base-neutrals and extractables: 50-150% recovery
- Organic chemistry-Acids: 25-125% recovery
- Other analysis: 75-125% recovery

Acceptable relative percent difference (RPD) of duplicate analyses is $\leq 20\%$ for duplicate results greater than 10 times the method detection limit (MDL). When one or both results are ≤ 10 times the MDL, the RPD acceptance range is $\leq 67\%$.

Acceptance limits will be updated annually at a minimum, when 20 or more new values have been generated. The summary of acceptance limits is revised and distributed to the appropriate lab groups after each update.

9.5 Reporting Limits

Reporting limits are generated based on a factor of 2-5 times the calculated MDL. Reporting limits are available to the client by request.

10. Data Collection, Reduction, and Reporting

10.1 Purpose and Applicability

This section defines the TraceAnalysis procedures for data collection, reduction, entry into the LIMS, validation, and reporting. All data is collected, reduced, entered, validated, and reported in accordance with this procedure unless an alternate scheme is outlined in a project-specific plan.

10.2 Responsibilities

10.2.1 Analysts

Analysts conduct data collection and reduction in accordance with this procedure.

10.2.2 Supervisors

Laboratory Supervisors review data, assist in corrective action procedures.

10.2.3 Quality Assurance

The QA Department reviews laboratory raw data and quality control data.

10.2.4 Director

The Laboratory Director reviews and signs final reports before sending them to clients.

10.3 Data Collection

10.3.1 Sample Preparation and Analysis

Sample preparation and analytical activities are documented in sufficient detail to allow the analysis to be recreated. The information must be recorded in a laboratory notebook or on preprinted worksheets, or retrievable from instrument output. This includes the following at a minimum:

• The analytical activity being performed (i.e. the specific analytical method or preparation method performed)

- The person(s) performing the activity and the date and time that the activity was initiated.
- Instrument parameters, including instrument identification and settings. Instrument settings may be referenced to previous documentation of instrument parameters.
- The analytical sequence must be documented (i.e. the chronological order of analysis). The following data for each sample, standard, and QC check ran in the analytical sequence must be recorded and/or retrievable from an instrument printout (quantitation report, etc.)
- QC sample type identification if QC sample
- Dilution
- Sample aliquot/final volume
- Instrument reading
- Units for all variables are specified, preferably in column headings
- TraceAnalysis sample number
- Final result
- Percent recovery and RPD
- The calibration curve from which data are quantified, identified by instrument and date ran, or by reference to a notebook and page number or a filename, if the initial calibration is included in the analytical run.
- Identification of the source of standards used for calibration, calibration verification, lab control samples, and matrix spikes referenced to a standards prep notebook and page number.
- Notes regarding any anomalies (e.g. change in color, formation of precipitate, sample foaming) or difficulties (e.g. instrument malfunction) encountered during analysis.
- The notebook identification number on each page.

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10.3.2 Data Recording and Error Correction

All handwritten data must be recorded using indelible ink. When an error in any hardcopy documentation of data is corrected, the person making the correction draws a single line through the erroneous data so as not to obscure the original entry. He/she then writes his/her initials, the date, and the correct information, if applicable, adjacent to the error.

10.4 Data Reduction

10.4.1 Qualitative Identification

Qualitative identification of organic compounds is performed according⁻to retention time matching. Second column confirmation by GC is performed upon request or when specified by the requested method.

10.4.2 Quantitation

The equations used to calculate final results are specified in the appropriate laboratory methods and SOPs. In general, the following rules concerning blank correction, reporting limits, significant figures, and rounding rules apply to those calculations.

10.4.2.1 Blank Correction

Blank correction is employed only when permissible on analytical procedures or methods performed by TraceAnalysis (e.g. titrations).

10.4.2.2 Significant Figures and Rounding Rules

All calculation results are rounded to the correct number of digits (usually 2) as the final calculation step. No result is rounded before reaching the final answer, even in a lengthy calculation.

To round a number, first determine the number of digits to be reported (the reportable figures, usually 2). Determine whether the digit to the immediate right of the right-most reportable figure is greater than, equal to, or less than 5. Ignore any digits further to the right unless the number is 5. If the number is greater than 5, round up (e.g. 1.66 is rounded to 1.7). If the number is less

than 5, truncate after the last reportable figure (e.g. 1.64 is rounded to 1.6). If the number is 5 and is followed by other non zero digits, then we add one to the preceding digit (e.g. 1.653 and 1.6501 will both round to 1.7). If the digit after the point of round off is a 5 and no other digits follow the 5, then we drop the 5 if the preceding digit is even and add one to the preceding digit if it is odd (e.g. 1.65 is rounded to 1.6 and 1.75 is rounded to 1.8).

Round results at the end of calculations to two digits as follows, with the exceptions noted:

- If the initial concentration of the sample is less than the reporting limit, express the reporting limit as 2 digits (e.g. <1.0 mg/L)
- If the initial concentration of the sample is above the reporting limit, and if expressed in scientific notation its exponent would be equal to that of the reporting limit expressed in scientific notation, report the result to 2 digits. (For example, if the reporting limit is 1.0 mg/L, the initial concentration is 5.148 mg/L and no dilutions were made, report the result as 5.1 mg/L.)
- If the initial concentration is above the reporting limit, and is expressed in scientific notation its exponent would be greater than that of the reporting limit expressed in scientific notation, report the result to 2 digits. (For example, if the reporting limit is 1.0 mg/L, and the initial concentration is 51.480 mg/L and no dilutions were made, report the result as 51 mg/L.)

Note: for quality control checks and PE samples, express results, recoveries, and relative percent differences using at lest three significant figures whenever possible.

10.4.3 Evaluation

The quality control data for each batch or analytical run are evaluated against acceptance limits. Whenever a quality control result exceeds acceptance limits, corrective action is required before turning in data for the batch or analytical run for data review. Corrective actions are recorded in the analysis log and on a nonconformance/corrective action form.

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10.5 Data Validation

10.5.1 Data Review

Following data reduction and before data entry, the raw data associated with the analytical run analysis log, instrument output (quantitation reports, chromatograms, spectra), calibration curves, etc. are forwarded to the Quality Assurance Officer for data review. The review encompasses the correctness, acceptability, and completeness of the following elements of data generation and handling. (All elements are not applicable to all tests.)

- Instrument tuning
- Initial Calibration
- Continuing calibration/calibration verification
- Calibration Blanks
- Method or preparation blanks
- Surrogate and/or lab control sample recovery
- Qualitative identifications
- Quantitation, including units and reportable figures
- Precision of duplicates
- Recovery of matrix spikes
- Holding Times
- Data qualifiers
- Data Entry

When unacceptable calibration or quality control check is generated, the data reviewer ensures that appropriate corrective action was taken before approving the data. Any defects are corrected. Raw data are corrected as necessary. If corrective action cannot be taken, the samples are qualified appropriately.

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Upon approval of the data, the reviewer initials the lab notebook, page(s), worksheet(s), or instrument printout, and indicates approval of the data, which allows the data to proceed to data entry and final report generation. Following data review and approval, preliminary results may be provided to the client when necessary. The results must be clearly labeled as being preliminary and subject to change upon completion of laboratory review.

10.5.2 Data Entry

Following data evaluation and review, the sample results and QC data are entered into the LIMS system by data entry personnel.

10.5.3 Data Reporting

Following data review, data are available for report preparation through the LIMS. The report consists of a lab analysis report and a quality control report. On most reports, the QC information is available on the same page as the lab analysis report. The lab analysis report contains the following information:

- A title, e.g. "Test Report"
- Name and address of laboratory
- An Order Identification Number
- Name and address of client
- Project information
- Matrix identification
- Characterization and condition of the test item
- Date sampled and date of sample receipt
- Date of performance of tests
- Identification of test method used

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- A percentage of certainty of test result
- Result and units for each analyte for each sample
- Any comments about the sample or results (e.g. matrix effect)
- A signature and title of person accepting responsibility for the content of the report

If requested by the client, results from organics analyses which fall between the method detection limit and the reporting limit may be reported with a flag indicating that the result is an estimate. Additionally, results quantitated over the standard range will also be reported with a flag indicating that the result is an estimate.

10.5.4 Quality Control Reporting

The quality control report contains the following information as applicable to the analyses:

- Supplemental information, including method reference, date and time of preparation (if applicable) and analysis, analyst
- Surrogate standard recoveries
- Method blank results
- Matrix spike and duplicate or matrix spike duplicate results

If any quality control sample result does not meet the applicable acceptance criteria, a footnote or comment will be included with the result in order to explain the nonconformance and corrective action taken, if appropriate. The quality control report may be further supplemented with initial and continuing calibration data and/or raw data upon request.

10.5.5 Final Report Review

After the LIMS report is printed, it is forwarded, with any supplemental information, including reviewed data sheets, to the Laboratory Director, who compiles and reviews the report, ensuring that all deliverables are correct. Errors and inconsistencies that are not evident in the initial review may become apparent when each result is evaluated in light of the results obtained for the other parameters: Specifically the following are observed:

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- Units and reportable figures
- Interparametric relationships (e.g. TDS/specific conductance, TOC/BOD/COD, dissolved/total, anion/cation balance, where appropriate)
- Reasonableness of results given the available information about the sample
- Method references
- Any problems with the data must be corrected before the final report is approved

10.6 Records Retention

All records of raw data, audits, quality control, and laboratory procedures are maintained in various files and notebooks throughout the lab. At the end of each year they are transferred to storage and kept five years.

All records of client sample analysis are maintained in client files. These records include copies of the original signed results, a copy of the chain-of-custody, and all raw data associated with the results. These records are transferred to storage at the end of the year and are retained for five years.

10.7 Confidentiality of Deliverables

The laboratory delivers the reports to specified contact listed on the chain-of-custody accompanying the samples. Where clients require transmission of test results by telephone, facsimile or other electronic or electromagnetic means, staff will follow documented procedures that ensure that the requirements are met and that confidentiality is preserved.

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11. Corrective Action

11.1 Initiation and Completion of Correction

If, because of an audit or QC sample analysis, a system defect is discovered, corrective action is implemented. The analyst, section supervisor, Quality Assurance Officer, or Laboratory Director may initiate the action and will participate in the corrective action. If previously reported data are affected by a situation requiring correction, the matter will be acted upon by the Quality Assurance Officer and Laboratory Director.

The steps that may compromise a closed-loop corrective action system are as follows:

- Define the problem
- Assign responsibilities for problem investigation
- Investigate and determine the cause of the problem
- Check all calculations
- Re-analyze the sample
- Verify the integrity of the spiking solution, laboratory control sample, or calibration standard
- Check instrument and operating conditions to preclude the possibility of malfunctions or operator error
- Determine the corrective action(s) necessary to eliminate the problem
- Assign and accept responsibilities for implementing the corrective action
- Establish the effectiveness of the corrective action and implement the correction
- Verify and document that the corrective action has eliminated the problem, using a nonconformance/corrective action report form. See figure 11-1.

Depending on the nature of the problem, the corrective action implemented may be formal or informal. In either case, occurrence of the problem, the corrective action employed, and verification that the problem has been eliminated must be documented.

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Figure 11-1 Nonconformance/Corrective Action

TRACEANAI NONCONFORM	LYSIS, INC Lubbock, TX Report ID # IANCE/CORRECTIVE ACTION EPORT FORM
PART I : ORIGINATOR	DATE/TIME:
ANALYST:	
DEPT./TEST:	PREP DATE:
PREP. ANALYST:	
DESCRIPTION OF NONCONFORMANCE:	
CORRECTIVE ACTION	· · · ·
	·
REPORTED BY:	DATE:
PART 2: DEPARTMENT SUPERVISOR	
SUPERVISOR'S COMMENTS OR RECOMMEN	NDATIONS:
APPROVED BY:	DATE:
PART 3: QA DEPARTMENT	
QA DEPARTMENT'S COMMENTS:	
NC/NA CLOSED BY:	DATE:
FORWARD ORI COPIES TO CL	IGINAL TO NC/CA LOGBOOK IENT'S FILE AND RAW DATA

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12. Performance Evaluations and System Audits

12.1 Internal Audits

The records, logbooks, and data files of each laboratory department are audited annually, at a minimum, by the Quality Assurance Department. The data is reviewed for completeness, accuracy, and adherence to standard operating procedures. Random project files are evaluated for compliance to procedures throughout the analytical process. There is review of all logbooks and records to ensure appropriate documentation of analyses are being recorded in the proper manner. For any deficiencies generated during the internal audit, the Managing Director will have the responsibility of responding to the deficiencies within 30 days and implementing any necessary corrective actions. If the internal audit uncovers data of doubtful quality, the laboratory will take immediate corrective action and will notify, in writing any client whose work has been affected.

12.2 External Audits

TraceAnalysis is audited as required by regulatory agencies to maintain laboratory certifications and approvals. Commercial clients with laboratory audition programs typically conduct on-site audits and perform data audits on a project-specific basis. These audits are conducted by the client or a consulting firm specializing in this service and operating under contract to the client.

12.3 Performance Evaluations

TraceAnalysis participates in the USEPA semi-annual wastewater (WP series) Performance Evaluation Studies. Trace also participates in client-sponsored performance evaluations by analyzing QC samples prepared and submitted by commercial clients in conjunction with their own QA program.

13. Assessment of Precision, Accuracy, Completeness, Representativeness, and Comparability

13.1 Accuracy

Accuracy is indicated by the measure of the difference between observed and true values. A minimum of one of every 20 environmental samples for organic analyses or one in 10 for inorganic analyses is spiked with a standard solution to assist in the evaluation of accuracy of the method for a given sample matrix through calculation of percent recovery of the matrix spike.

Each batch of up to 20 samples is prepared with a laboratory control sampled to ensure the analysis system is operating in control. The percent recovery for the LCS is calculated by comparison of the value obtained for the analysis with the true value for the LCS.

Surrogate compounds are spiked into samples analyzed by GC and GC/MS methods. The percent recoveries of the surrogates are used as an indicator of the accuracy and extraction efficiency of the analysis.

The calculation of percent recovery is performed in the following manner:

Matrix Spike Reco	overy
% Recover	$y = \underline{SSR} - \underline{USR} \times 100\%$
	SA
Where:	SSR = Spiked sample result
	USR = Unspiked sample result
	SA = Spike added

Surrogate or Lab Control Sample Recovery % Recovery = <u>Result obtained</u> x 100% True Value

13.2 Precision

Precision refers to the reproducibility of results obtained for the analyses of duplicate samples or matrix spiked duplicate samples. One out of every 20 samples of similar

matrix analyzed by each method for organics (1 in 10 for inorganics) is run in duplicate or as matrix spike duplicates for determining precision.

The results of the duplicate analyses are computed and the absolute relative percent difference (RPD) is calculated as follows:

$$\frac{\text{RPD}}{\frac{1}{2}(\text{R1} - \text{R2})} \times 100\%$$

where: R1 = First replicate result

R2 = Second replicate result

The RPD must fall within set acceptance limits for the results to be accepted and subsequent data validated.

13.3 Completeness

Data completeness can be quantified during data assessment. A statement of expected completeness for a project is one data quality objective. TraceAnalysis is able to provide data, meeting QC acceptance criteria, for 95% or more of the requested determinations. It is important for planners to identify any sample types, such as control or background locations, which require 100% completeness.

13.4 Representativeness

Representativeness is a qualitative element that is related to the ability to collect a sample that reflects the characteristics of that part of the environment that is to be assessed. Sample representativeness is dependent on the sampling techniques used and is considered individually for each project. The laboratory recognizes its role in achieving within-sample representativeness and uses appropriate techniques to obtain a representative aliquot. Some tests are performed multiple times if obtaining a representative aliquot is not possible due to heterogeneity or small sample size.

13.5 Comparability

The objective of comparability is to produce results that do not differ significantly form those produced by other parties for the same purpose. TraceAnalysis uses SOPs based on EPA-approved methods in order to achieve comparability with data from previous studies and from other laboratories. SOPs are written to incorporate the method requirements. TraceAnalysis participates in external and inter-laboratory perfromance evaluation (PE) studies as an additional means of establishing comparability in the laboratory.

14. Quality Assurance Reporting

14.1 Reports to Director

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Through quality reports to the Laboratory Director, it is ensured that management personnel are informed of situations, which could affect the performance of the laboratory. Reports are provided by the Quality Assurance Officer to the Laboratory Director. This report addresses the quality assurance activities including details of corrective actions implemented, audit results, and QC summary information. In addition to the QA reports, weekly meetings are used to communicate to the laboratory's management staff pertinent information related to QA/QC issues.

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15. Training

15.1 Introduction

This section of the QA Plan describes the TraceAnalysis program for training in areas where quality is affected. Specific areas where training must be documented include:

- Analytical methods training
- Quality assurance/quality control (QA/QC) training
- Safety Training

Other types of training also occur but are not at this time required to meet the requirements of the quality assurance program; these include such subjects as computer training, continuing education, and seminars.

15.2 Analytical Methods Training

All analysts are trained and supervised in performing specific analytical procedures before working unsupervised. The laboratory section supervisors are responsible for training within their work groups. A supervisor or senior analyst typically conducts the training, using method-specific analytical SOPs as training guides.

A training record is used to document the trainee's proficiency in performing the procedure. For some methods, analyst proficiency is also demonstrated through the analysis of standard materials, with documentation retrievable from the lab notebook and raw data.

Each section supervisor will determine the frequency of retraining, based on revisions to the SOPs or the methods themselves.

15.3 QA/QC Training

The Quality Assurance Officer (QAO) conducts training of new hires in general QA/QC principles. The QAO determines the frequency of retraining, based on deficiencies determined during perfromance evaluation or systems audits. Additionally, the QAO may provide project-specific training before the laboratory analyzes samples for a major project or a project with specific QA/QC or analytical requirements.

A training record is used to document each trainee's attendance at a given training session.

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15.4 Safety Training

The laboratory Safety Officer conducts training of new hires in the TraceAnalysis safety program and Chemical Hygiene Plan. Annually all employees are given safety training, which includes the following subjects:

- Safety and Chemical Hygiene Plan
- Fire Safety

In addition, selected employees are trained annually in respirator use, waste handling/hazardous materials, and/or first aid/CPR. The Safety Officer also conducts other safety-related training as needs arise. A training record is used to document each trainee's attendance at a given training session.

15.5 Training Records

Records are maintained documenting each employee's training in analytical methods, QA/QC principles, and safety. Training records shall specify the trainee, trainer, date, and subject of the training session. The results of proficiency testing, where applicable, should also be included.

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Appendix 1 Quality Manual Revision Log

Revision Description	Section #	Date	Authorized Signature
Input of a2La guidelines	1,2,4,6,	Res # 4	16
Correction of Errors	7.8.10.13	08 Dec 97	famer ayer
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8021B LCS Control Limits % Recovery

Water

Compound	Control Limits
MTBE	82-124
Benzene	78-125
Toluene	80-124
Ethyl Benzene	84-121
Xylens	88-124

Soil

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Compound	Control Limits
MTBE	69-126
Benzene	77-118
Toluene	72-114
Ethyl Benzene	75-120
Xylens	68-124

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8021B Surrogates Control Limits % Recovery

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Water

Compound TFT 4-BFB <u>Control Limits</u> 77-113 86-120

Soil

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. ₹-3 Compound TFT 4-BFB

<u>Control Limits</u> 68-126 67-129

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8260B LCS Control Limits % Recovery

Water

Compound	<u>Control Limits</u>
MTBE	69-122
Benzene	76-121
Toluene	76-118
Ethyl Benzene	71-123
Xylens	73-122

Soil

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Compound	Control Limits
MTBE	54-112
Benzene	67-117
Toluene	69-114
Ethyl Benzene	69-114
Xylens	69-114
Benzene Toluene Ethyl Benzene Xylens	67-117 69-114 69-114 69-114

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8260B Surrogate Control Limits % Recovery

Surrogate	Soil	Water	TCLP
4-Bromofluorobenzene*	74-121	86-115	86-115
Dibromofluorobenzene*	80-120	86-118	86-118
Toluene*	81-117	88-110	88-110

*Table 8, SW-846-8260A.

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TCLP Volatiles Control Limits % Recovery

Compounds	Control Limits
1,1-Dichloroethane	74-133
1,2-Dichloroethane	75-124
1,4-Dichloroethane	73-122
Benzene	76-123
Carbon Tetrachloride	82-130
Chlorobenzene	79-122
Chloroform	79-125
MEK	67-135
Tetrachloroethene	81-126
Trichloroethene	76-127
Vinyl Chloride	53-134

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8270C LCS Control Limits % Recovery

Soil

Compound	Control Limits
1,2,4-Trichlorobenzene	6-113
1,4-Dichlorobenzene	0-100
2,4-Dinitrotoluene	19-115
2-Chlorophenol	2-102
4-Chloro-3-Methylphenol	5-116
4-Nitrophenol	D*
Acenaphthene	10-116
n-Nitrosodi-n-propylamine	3-120
Pentachlorophenol	D*
Phenol	0-101
Pyrene	46-152

Water

<u>Compound</u>	Control Limits
1,2,4-Trichlorobenzene	44-125*
1,4-Dichlorobenzene	20-116*
2,4-Dinitrotoluene	39-134*
2-Chlorophenol	23-126*
4-Chloro-3-Methylphenol	22-122*
4-Nitrophenol	0-86*
Acenaphthene	47-132*
n-Nitrosodi-n-propylamine	9-138
Pentachlorophenol	19-123
Phenol	5-92*
Pyrene	54-115*

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SW-846 Surrogate Limits D = Detectable

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8270C Surrogate Control Limits % Recovery

			PAH	PAH
<u>Soil</u>	Water	TCLP	Water	<u>Soil</u>
21-100	25-121	21-100		
10-94	24-113	10-94		
35-114	23-120	35-114	35-114	26-120
43-116	30-115	43-116	43-116	30-115
10-115	20-104	15-122		
35-141	28-137	33-141	48-141	77-137
	<u>Soil</u> 21-100 10-94 35-114 43-116 10-115 35-141	SoilWater21-10025-12110-9424-11335-11423-12043-11630-11510-11520-10435-14128-137	SoilWaterTCLP21-10025-12121-10010-9424-11310-9435-11423-12035-11443-11630-11543-11610-11520-10415-12235-14128-13733-141	SoilWaterTCLPWater21-10025-12121-10010-9410-9424-11310-9435-11423-12035-11443-11630-11543-11643-11620-10415-12235-14128-13733-14148-141

8270C Surrogate Limits are from default values of Table 8, 8270, SW-846

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PAH LCS Control Limits % Recovery

Soil

Compound	Control Limits
Acenapthene	28-96
Acenapthylene	36-94
Anthracene	56-99
Benzo(g,h,i)Perylene	60-112
Benzo(a)Anthracene	72-124
Benzo(a)Pyrene	29-137
Benzo(b)Fluoranthene	53-125
Benzo(k)Fluoranthene	66-145
Chrysene	59-137
Dibene(a,h)Anthracene	59-106
Fluoranthene	65-119
Fluorene	42-100
Indeno(1,2,3-cd)Pyrene	65-111
Napthalene	28-81
Pyrene	72-126

Water

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Compound	Control Limits
Acenapthylene	33-115*
Anthracene	55-104
Benzo(g,h,i)Perylene	53-107
Benzo(a)Anthracene	68-115
Benzo(a)Pyrene	62-111
Benzo(b)Fluoranthene	55-110
Benzo(k)Fluoranthene	65-136
Chrysene	51-133
Dibene(a,h)Anthracene	49-101
Fluoranthene	68-116
Fluorene	59-121*
Indeno(1,2,3-cd)Pyrene	54-109
Napthalene	21-111
Phenanthrene	67-112
Pyrene	69-115*
Acenapthene	47-128*

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*SW-846 Limits

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TCLP Semi-volatiles LCS Control Limits % Recovery

<u>Compounds</u>	Control Limits
1,4-Dichlorbenzen	20-71*
2,4,5-TP	16-115
2,4,5-Trichlorophenol	37-96*
2,4,6-Trichlorophenol	10-94
2,4-D	14-101
2,4-Dinitrotoluene	39-100*
Hexachloroethane	40-79*
Hexachlorobenzene	56-103
Hexachlorobutadiene	24-83*
m,p-Cresol	13-69
Nitrobenzene	35-88*
o-Cresol	17-74
Pentachlorophenol	46-101
Pyridine	D*

Limits from Table 6, Method 8270B. D = Detectable

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Metals MS/MSD Control Limits % Recovery

Compounds

Control Limits

Metals 6010B

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75-125*

*Limits for all 6010B metals are set from SW-846.

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Wet Chemistry MS/MSD Control Limits % Recovery

COMPOUND	<u>Control Limits</u>
Cyanide	62-119
Nitrate	65-129
Ammonia	70-114
Fluoride	66-124
Phosphate	61-132
Sulfate	72-136
Phenolics	67-137
TKN-N	66-115
Chloride	85-112
Fluoride	75-117
Nitrate	93-110
Nitrite	85-110
Sulfate	89-114

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BTEX Method Detection Limits and Reporting Limits Water

BTEX 8021B	MDL	RL
<u>GC-1</u>	<u>(ppb)</u>	<u>(ppb)</u>
MTBE	0.19	1
Benzene	0.16	1
Toluene	0.11	1
Ethyl Benzene	0.16	1
Xylene	0.19	3
Styrene	0.15	1

BTEX 8021B	MDL	RL
<u>GC-1</u>	<u>(ppb)</u>	<u>(ppb)</u>
MTBE	0.39	1
Benzene	0.3	1
Toluene	0.31	1
Ethyl Benzene	0.24	1
Xylene	0.81	3
Styrene	0.15	1

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BTEX Method Detection Limits and Reporting Limits

Soil

BTEX 8021B	MDL	RL
<u>GC-1</u>	<u>(ppb)</u>	<u>(ppb)</u>
MTBE	22	50
Benzene	23	50
Toluene	22	50
Ethyl Benzene	24	50
Xvlene	76	150

BTEX 8021B	MDL	RL
<u>GC-1</u>	<u>(ppb)</u>	<u>(ppb)</u>
MTBE	13	50
Benzene	3.6	50
Toluene	8.3	50
Ethyl Benzene	3.8	50
Xylene	45	150

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8015 Gasoline Method Detection Limit and Reporting Limit

Compound	MDL	RL
	<u>(ppm)</u>	<u>(ppm)</u>
8015 Gasoline	1.63	10

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8260B Method Detection Limits and Reporting Limits Water

	MDL	RL
Compounds	(ppb)	(ppb)
Dichlorodifluoromethane	0.50	1
Chloromethane	0.39	. 1
Vinyl Chloride	0.27	2
Bromomethane	0.22	5
Chloroethane	0.44	1
Trichlorofluoromethane	0.42	1
1,1-Dichloroethene	0.38	1
Methylene Chloride	1.77	5
trans-1,2-Dichloroethene	0.22	1
1,1-Dichloroethene	0.21	1
cis-1,2-Dichloroethene	0.20	1
Chloroform	0.18	1
2,2-Dichloropropane	0.32	1
Bromochloromethane	0.18	1
1,2-Dichloroethane	0.19	1
1,1,1-Trichloroethane	0.31	1
Carbon Tetrachloride	0.45	1
1,1-Dichloropropene	0.23	1
Benzene (M)	0.18	1
1,2-Dichloropropane (C)	0.18	1
Trichloroethene (M)	0.31	1
Dibromomethane	0.23	1
Bromodichloromethane	0.11	1
cis-1,3-Dichloropropene	0.11	1
trans-1,3-Dichloropropene	0.08	1
Toluene (C,M)	0.15	1
1,1,2-Trichloroethane	0.14	1
1,3-Dichloropropane	0.10	1
Dibromochloromethane	0.21	1

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8260B Method Detection Limits and Reporting Limits Water

	MDL	RL
Compounds	(ppb)	(ppb)
1,2-Dibromoethane	0.15	1
Tetrachloroethene	0.35	1
Chlorobenzene (C,M,P)	0.20	1
1,1,1,2-Tetrachloroethane	0.29	1
Ethylbenzene (C,M,P)	0.22	1
m&p-Xylene	0.44	1
Bromoform	0.34	1
Styrene	0.14	1
o-Xylene	0.17	1
1,1,2,2-Tetrachloroethane (P)	0.10	1
1,2,3-Trichloropropane	0.14	1
Isopropylbenzene	0.16	1
Bromobenzene	0.14	1
2-Chlorotoluene	0.14	1
n-Propylbenzene	0.17	1
4-Chlorotoluene	0.21	1
1,3,5-Trimethylbenzene	0.15	1
tert-Butylbenzene	0.15	1
1,2,4-Trimethylbenzene	0.19	1
1,4-Dichlorobenzene	0.48	2
sec-Butylbenzene	0.18	1
1,3-Dichlorobenzene	0.12	2
4-Isopropyltoluene	0.19	1
1,2-Dichlorobenzene	0.17	2
n-Butylbenzene	0.17	1
1,2-Dibromo-3-Chloropropane	1.22	5
1,2,3-Trichlorobenzene	0.38	5
Napthalene	0.23	1
1,2,4-trichlorobenzene	1.00	5
Hexachlorobutadiene	1.35	5

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8260B Method Detection Limits and Reporting Limits Soil

	MDL	RL
Compounds	(ppb)	(ppb)
Dichlorodifluoromethane	12.5	25
Chloromethane (P)	9.75	25
Vinyl Chloride (C)	6.75	50
Bromomethane	5.5	25
Chloroethane	11	25
Trichlorofluoromethane	10.5	25
1,1-Dichloroethene (C,M)	9.5	25
Methylene Chloride	44.25	125
trans-1,2-Dichloroethene	5.5	25
1,1-Dichloroethene (P)	5.25	25
cis-1,2-Dichloroethene	5	25
Chloroform (C)	4.5	25
2,2-Dichloropropane	8	25
Bromochloromethane	4.5	25
1,2-Dichloroethane	4.75	25
1,1,1-Trichloroethane	7.75	25
Carbon Tetrachloride	11.25	25
1,1-Dichloropropene	5.75	25
Benzene (M)	4.5	25
1,2-Dichloropropane (C)	4.5	25
Trichloroethene (M)	7.75	25
Dibromomethane	5.75	25
Bromodichloromethane	2.75	25
cis-1,3-Dichloropropene	2.75	25
trans-1,3-Dichloropropene	2	25
Toluene (C,M)	3.75	25
1,1,2-Trichloroethane	3.5	25
1,3-Dichloropropane	2.5	25
1,1,1,2-Tetrachloroethane	7.25	25
Ethylbenzene (C)	5.5	25

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8260B Method Detection Limits and Reporting Limits Soil

	MDL	RL
Compounds	(ppb)	(ppb)
m&p-Xylene	11	25
Bromoform(P)	8.5	25
Styrene	3.5	25
o-Xylene	4.25	25
1,1,2,2-Tetrachloroethane (P)	2.5	25
1,2,3-Trichloropropane	3.5	25
Isopropylbenzene	4	25
Bromobenzene	3.5	25
2-Chlorotoluene	3.5	25
n-Propylbenzene	4.25	25
4-Chlorotoluene	5.25	25
1,3,5-Trimethylbenzene	3.75	25
tert-Butylbenzene	3.75	25
1,2,4-Trimethylbenzene	4.75	25
1,4-Dichlorobenzene	12	50
sec-Butylbenzene	4.5	25
1,3-Dichlorobenzene	3	50
4-Isopropyltoluene	4.75	25
1,2-Dichlorobenzene	4.25	50
n-Butylbenzene	4.25	25
1,2-Dibromo-3-Chloropropane	30.5	125
1,2,3-Trichlorobenzene	9.5	125
Napthalene	5.75	25
1,2,4-trichlorobenzene	25	125
Hexachlorobutadiene	33.75	125

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8270C Method Detection Limits and Reporting Limits Water

	MDL	RL
Compounds	(ppm)	(ppm)
n-Nitrosodmethylamine	0.00044	0.001
2-Picoline	0.00138	0.001
Methyl Methanesulfonate	0.00049	0.001
Ethyl Methanesulfonate	0.00065	0.001
Phenol	0.00036	0.001
Aniline	0.00095	0.005
bis(2-Chloroethyl)ether	0.00054	0.005
2-Chlorophenol	0.00068	0.005
1,3-Dichlorobenzene	0.00057	0.001
1,4-Dichlorobenzene	0.00065	0.001
Benzly Alcohol	0.00057	0.005
1,2-Dichlorobenzene	0.00067	0.001
2-Methylphenol	0.00061	0.001
bis(2-Chloroisopropyl)ether	0.00072	0.005
4-Methylphenol	0.00064	0.001
Acetophenone	0.00074	0.005
n-Nitrosodi-n-propylamine	0.00078	0.001
Hexachloroethane	0.00056	0.001
Nitrobenzene	0.00076	0.001
n-Nitrosopiperidine	0.00079	0.005
Isophorene	0.00085	0.005
2-Nitrophenol	0.00059	0.005
2,4-Dimethylphenol	0.00077	0.005
bis(2-Chloroethoxy)methane	0.00083	0.001
2,4-Dichlorophenol	0.00078	0.005
1,2,4-Trichlorobenzene	0.00063	0.001
Benzoic Acid	0.00481	0.01
Napthalene	0.00066	0.001
a,a-Dimethylphenethylamine	0.00944	0.01
4-Chloroaniline	0.00075	0.005
2,6-Dichlorophenol	0.00083	0.005
Hexachlorobutadiene	0.00060	0.001
n-Nitroso-di-n-butylamine	0.00078	0.005
4-Chloro-3-methylphenol	0.00077	0.005

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8270C Method Detection Limits and Reporting Limits Water

	MDL	RL
Compounds	(ppm)	(ppm)
2-Methynanthalene	0.00068	0.001
1.2.4.5 Tetrachlorobenzene	0.0008	0.001
Hexachlorogyglopontadiono	0.00003	0.001
2.4.6 Trichlorophenol	0.00232	0.005
2,4,0-Themorophenol	0.00072	0.005
2,4,5-1 Hemorophenor	0.00083	0.003
	0.0003	0.001
2 Nitroopilino	0.00073	0.001
Dimothylabthalata	0.00007	0.003
	0.00093	0.001
2 6 Divitestaluces	0.00083	0.001
2,0-Dimitrotoluene	0.00073	0.001
	0.0008	0.005
Acenaphtnene	0.00079	0.001
2,4-Dinitrophenol	0.02013	0.025
Dibenzoturan	0.00082	0.005
Pentachlorobenzene	0.00093	0.001
4-Nitrophenol	0.00164	0.005
2,4-Dinitrotoluene	0.00083	0.001
2,3,4,6-Tetrachlorophenol	0.00078	0.005
2-Naphthylamine	0.00050	0.005
Fluorene	0.00084	0.001
4-Chlorophenyl Phenylether	0.00101	0.001
Diethylphthalate	0.00096	0.001
4-Nitroaniline	0.00053	0.005
4,6-Dinitro-2-Methylphenol	0.00444	0.005
n-Nitrosodiphenylamine	0.00087	0.001
Diphenylhydrazine	0.00082	0.005
1-Naphthylamine	0.00083	0.005
4-Bromophenyl Phenylether	0.00105	0.001
Phenacetin	0.00021	0.005
Hexachlorobenzene	0.00093	0.001
4-Aminobiphenyl	0.00034	0.005
Pentachlorophenol	0.00080	0.005
Anthracene	0.00090	0.001

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8270C Method Detection Limits and Reporting Limits Water

	MDL	RL
Compounds	(ppm)	(ppm)
Benzidine	0.01034	0.01
Pyrene	0.00033	0.001
p-Dimethylaminoazobenzene	0.00108	0.001
Butylbenxylphthalate	0.00024	0.001
Benz[a]anthracene	0.00037	0.001
3,3-Dichlorobenzidine	0.00050	0.001
Chrysene	0.00058	0.001
bis(2-Ethylhexyl)phthalate	0.00278	0.005
Di-n-Octylphthalate	0.00047	0.001
Benzo[b]fluoranthene	0.00073	0.001
Benzo[k]fluoranthene	0.00108	0.001
7,12-Dimethylbenz[a]anthracene	0.00075	0.001
Benzo[a]pyrene	0.00038	0.001
3-Methylcholanthracene	0.00033	0.001
Dibenzo[a,j]acridine	0.00048	0.001
Indeno[1,2,3-cd]pyrene	0.00060	0.001
Dibenz[a,h]anthracene	0.00050	0.001
Benzo[g,h,i]perylene	0.00061	0.001

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8270C Method Detection Limits and Reporting Limits Soil

	MDL	RL
Compounds	(ppm)	(ppm)
n-Nitrosodmethylamine	0.043	0.25
2-Picoline	1 270	2.5
Methyl Methanesulfonate	0.064	0.25
Ethyl Methanesulfonate	0.076	0.25
Phenol	0,109	0.25
Aniline	0.411	1.25
bis(2-Chloroethyl)ether	0.085	1.25
2-Chlorophenol	0.082	1.25
1,3-Dichlorobenzene	0.056	0.25
1,4-Dichlorobenzene	0.095	0.25
Benzly Alcohol	0.069	1.25
1,2-Dichlorobenzene	0.078	0.25
2-Methylphenol	0.082	0.25
bis(2-Chloroisopropyl)ether	0.093	1.25
4-Methylphenol	0.073	0.25
Acetophenone	0.080	1.25
n-Nitrosodi-n-propylamine	0.071	0.25
Hexachloroethane	0.074	0.25
Nitrobenzene	0.066	0.25
n-Nitrosopiperidine	0.062	1.25
Isophorene	0.062	1.25
2-Nitrophenol	0.158	1.25
2,4-Dimethylphenol	0.057	1.25
bis(2-Chloroethoxy)methane	0.099	0.25
2,4-Dichlorophenol	0.082	1.25
1,2,4-Trichlorobenzene	0.089	0.25
Benzoic Acid	4.000	5
Napthalene	0.095	0.25
a,a-Dimethylphenethylamine	0.110	2.5
4-Chloroaniline	0.077	1.25
2,6-Dichlorophenol	0.097	1.25
Hexachlorobutadiene	0.083	0.25
n-Nitroso-di-n-butylamine	0.062	1.25

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8270C Method Detection Limits and Reporting Limits Soil

	MDL	RL
Compounds	(ppm)	(ppm)
4 Chloro 3 Mathylphanol	0.083	1 25
2 Methynanthalana	0.085	0.25
1.2.4.5-Tetrachlorobenzene	0.095	0.25
Hexachlorocyclopentadiene	0.160	0.25
2.4.6-Trichlorophenol	0.009	1.25
2.4.5-Trichlorophenol	0.091	1.25
2-Chloronanhthalene	0.051	0.25
1-Chloronaphthalene	0.130	0.25
2-Nitroaniline	0.150	1.25
Dimethylphthalate	0.005	0.25
Acenaphthylene	0.078	0.25
2 6-Dinitrotoluene	0.071	0.25
3-Nitroaniline	0.084	1.25
Acenaphthene	0.077	0.25
2 4-Dipitrophenol	0.274	1.25
Dibenzofiran	0.085	1.25
Pentachlorobenzene	0.074	0.25
4-Nitrophenol	0.071	1.25
2.4-Dinitrotoluene	0.088	0.25
2,3,4,6-Tetrachlorophenol	0.076	1.25
2-Naphthylamine	1.600	2.5
Fluorene	0.072	0.25
4-Chlorophenyl Phenylether	0.068	0.25
Diethylphthalate	0.087	0.25
4-Nitroaniline	0.041	1.25
4,6-Dinitro-2-Methylphenol	0.862	1.25
n-Nitrosodiphenylamine	0.051	0.25
Diphenylhydrazine	0.059	1.25
1-Naphthylamine	1.800	2.5
4-Bromophenyl Phenylether	0.075	0.25
Phenacetin	0.101	1.25
Hexachlorobenzene	0.086	0.25
4-Aminobiphenyl	0.097	1.25
Pentachlorophenol	0.107	1.25

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8270C Method Detection Limits and Reporting Limits Soil

	MDL	RL
Compounds	(ppm)	(ppm)
Anthracene	0.077	0.25
Pentachloronitrobenzene	0.085	1.25
Pronamide	0.103	0.25
Phenanthrene	0.087	0.25
Di-n-Butylphthalate	0.126	0.25
Fluoranthene	0.089	0.25
Benzidine	4.970	5
Pyrene	0.102	0.25
p-Dimethylaminoazobenzene	0.014	0.25
Butylbenxylphthalate	0.250	0.25
Benz[a]anthracene	0.128	0.25
3,3-Dichlorobenzidine	0.682	1.25
Chrysene	0.126	0.25
bis(2-Ethylhexyl)phthalate	0.080	0.25
Di-n-Octylphthalate	0.196	0.25
Benzo[b]fluoranthene	0.116	0.25
Benzo[k]fluoranthene	0.158	0.25
7,12-Dimethylbenz[a]anthracene	0.163	0.25
Benzo[a]pyrene	0.166	0.25
3-Methylcholanthracene	0.176	0.25
Dibenzo[a,j]acridine	1.360	2.5
Indeno[1,2,3-cd]pyrene	0.107	0.25
Dibenz[a,h]anthracene	0.126	0.25
Benzo[g,h,i]perylene	0.132	0.25

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TCLP Semi-Volatile Method Detection Limits and Reporting Limits

Compounds	MDL	RL
	(ppm)	(ppm)
Pyridine	0.00441034	0.25
1,4-Dichlorobenzene	0.00325476	1.25
2-Methylphenol	0.00303565	0.25
4-Methylphenol	0.00322427	0.25
Hexachloroethane	0.00277836	0.25
Nitrobenzene	0.00382116	0.25
Hexachlorobutadiene	0.00298959	5
2,4,6-Trichlorophenol	0.00358226	0.25
2,4,5-Trichlorophenol	0.00443269	0.25
2,4-Dinitrotoluene	0.00415553	0.25
2,4-D	0.00300793	0.25
Hexachlorobenzene	0.00465728	1.25
2,4,5-TP	0.01470939	0.25
Pentachlorophenol	0.00398153	0.25

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TPH Method Detection Limits and Reporting Limits Water

Compounds	MDL (ppm)	RL (ppm)
TPH, 418.1	0.29	0.50
TX1005, Gas		10
TX1005, Deisel		10

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	MDL	RL
Compounds	(ppm)	(ppm)
TPH, 418.1		10
TX1005, Gas		50
TX1005, Deisel		50

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ICP Method Detection Limits and Reporting Limits Water

	ARL Fison With Ultrasc	s 3560 AES onic Nebulzer	Leema	n DRE	Leema With Ultrasc	n DRE onic Nebulzer
Compounds	MDL (ppm)	RL (ppm)	MDL (ppm)	RL (ppm)	MDL (ppm)	RL (ppm)
As	0.005	0.1	0.019	0.050	0.0017	0.003
Se	0.010	0.1	0.034	0.10	0.0047	0.010
Cr	0.003	0.05	0.0057	0.010	0.0011	0.002
Cd	0.008	0.02	0.0015	0.005	0.0012	0.002
Pb	0.008	0.1	0.0067	0.010	0.0008	0.002
Ag	0.002	0.05	0.0023	0.005	0.0004	0.001
Ba	0.006	0.2	0.0016	0.005		
Sb	0.0082	0.1	0.011	0.020		
Be	0.0038	0.01	0.0007	0.002		
V	0.0065	0.05	0.0033	0.007		
Sr	0.053	0.1	0.0015	0.003		
Zn	0.0026	0.02	0.0016	0.003		
Al	0.017	0.2	0.0955	0.20		
Fe	0.0060	0.03	0.0058	0.010		
Со	0.0036	0.03	0.0015	0.003		
Ni	0.0056	0.01	0.0025	0.005		
Tl	0.028	0.7	0.0036	0.007		
Cu	0.0083	0.02	0.0099	0.020		
Мо	0.0051	0.1	0.0072	0.020		
Mn			0.0016	0.003		
Sn			0.0035	0.010		
Ca			0.0348	0.50		
Mg			0.0626	0.50		
Na			0.3045	1.0		
К			0.3686	1.0		
В			0.008	0.020		
Ti			0.014	0.050		

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ICP Method Detection Limits and Reporting Limits Soil

ARL Fisons 3560 AES With Ultrasonic Nebulzer Leeman DRE

Compounds	MDL (ppm)	RL (ppm)	MDL (ppm)	RL (ppm)
As	0.5	10	2.0	5
Se	1.0	10	4.8	10
Cr	0.3	5	1.0	5
Cd	0.8	2	0.9	2
Pb	0.8	10	2.5	5
Ag	0.2	5	0.9	2
Ba	0.6	20	1.3	5
Sb	0.8	10	1.3	5
Be	0.4	0.1	1.0	2
V	0.6	5	1.0	2
Sr	5.0	10	1.2	5
Zn	0.3	2	0.9	2
Al	2.0	20	5.7	10
Fe	0.6	3	1.7	3
Co	0.4	3	0.7	2
Ni	0.6	10	0.6	1
Tl	0.3	70	1.0	2
Cu	0.8	2	1.5	3
Мо	0.5	10	1.5	3
Mn			2.0	5
Sn			2.1	5

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PE 4100ZL Method Detection Limits and Reporting Limits Water

Compounds	MDL (ppb)	RL (ppb)
As	0.91	2
Se	1.0	2
Ag	0.23	0.5
Cd	0.62	1
Pb	0.57	1

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Murcury Method Detection Limit and Reporting Limit

Compounds	MDL (ppb)	RL (ppb)
Hg	0.16	1

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TOC Method Detection Limit and Reporting Limit

Compounds	MDL (ppm)	RL (ppm)
ТОС	1.23	2

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TOX Method Detection Limit and Reporting Limit Water

Compounds	MDL (ppm)	RL (ppm)
тох	4.2	5

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TOX Method Detection Limit and Reporting Limit

	Soil	
Compounds	MDL (ppb)	RL (ppb)
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Wet Chemistry Method Detection Limits and Reporting Limits

	MDL	RL
Compounds	(ppm)	(ppm)
A 11-01:		1.0
Alkalinity		1.0
Ammonia-N	0.66	1.0
Phenolics	0.013	0.02
TKN-N (Soil)	1.2	2.0
TKN-N (Water)	2.7	10
Cyanide, Amenable (Soil)	0.13	0.20
Cyanide, Total (Soil)	0.13	0.20
Fluoride 340-2	0.025	.10
Cyanide, Amenable	0.013	0.02
Cyanide, Total	0.013	0.02
Phosphate (Ortho-phosphorous) 365.2	0.001	0.010
COD	19	25
Fluoride 300.0	0.17	0.50
Chloride 300.0	1.5	5.0
Nitrite-N 300.0	0.18	0.50
Nitrate-N 300.0	0.17	0.50
Sulfate 300.0	1.61	5.0
TDS		10
Total Phenolics SM5530C	0.003	0.010
Chloride 4500 Cl-B	1.6	5.0
Hexavalent Cr	0.00039	0.010
Sulfides	0.40	1.0
Phosphate 300.0	0.30	1.0
Nitrate	0.094	0.20

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Oil & Grease Method Detection Limit and Reporting Limit

Compounds	MDL (ppm)	RL (ppm)
Oil & Grease	2.0	5.0

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LIST OF ACRONYMS/ABBREVIATIONS

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AA	Atomic Absorption
ACS	American Chemical Society
AES	Atomic emission spectroscopy
ASTM	American Society for Testing Materials
BFB	4-bromofluorobenzene
BNA	Base/neutral acid
BOD	Biological oxygen demand
CCC	Continuing calibration check
CCV	Continuing calibration verification
CFR	Code of Federal Regulations
CVAA	Cold vapor atomic absorption
DFTPP	Decafluorotriphenylphosphine
DQO	Data quality objectives
ECD	Electron capture detector
EOX	Extractable organic halogens
EPA	Environmental Protection Agency
FID	Flame ionization detector
FTIR	Fourier transform Infrared
GC	Gas chromatograph
GFAA	Graphite Furnace Atomic Absorption
GC/MS	Gas Chromatograph/mass spectroscopy
HDPE	High density polyethylene
ICP	Inductively coupled plasma
ICP-AES	Inductively coupled plasma/atomic emission spectroscopy
ICV	Initial calibration verification
ID	Identification
IDC	Initial demonstration of competence (samples)
IDL	Instrument detection limit
IPC	Instrument performance check
ISE	Ion-selective electrode
LCS	Laboratory control samples
LCSD	Laboratory control samples duplicate
LIMS	Laboratory information management system
MDL	Method detection limit
MS	Matrix spike
MSA	Multiple standard addition
MSD	Matrix spike duplicate
NBS	National Bureau of Standards (now NIST)
NC/CA	Non conformance/corrective action
ND	Non-detected
NIST	National Institute for Standards and Technology (formerly NBS)
NTIS	National Technical Information Service
PAH	Polynuclear aromatic hydrocarbons
PCB	Polychlorinated biphenyl
PDS	Post-digestion spikes
PE	Performance evaluation
PID	Photoionization detector

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QA	Quality assurance
QAO	Quality Assurance Officer
QAPjP or	· ·
QAPP	Quality assurance project plan
QC	Quality control
QCS	Quality control sample
%R	Percent recovery
RCRA	Resource Conservation and Recovery Act
RPD	Relative percent difference
RRF	Relative response factors
RRT	Relative retention time
RSD	Relative standard deviation
SOP	Standard operating procedures
SPCC	System performance check compounds
SPE	Solid phase extraction
SVO	Semivolatile organic
SVOC	Semivolatile organic compound
TAT	Turn-around time
TCL	Target compound list
TCLP	Toxicity characteristic leaching procedure
TICs	Tentatively identified compounds
TRPH	Total recoverable petroleum hydrocarbons
USEPA	United States Environmental Protection Agency
UV	Ultraviolet
VOA	Volatile organic analysis
VOC	Volatile organic compound
WP	Water Pollution (PE Sample)

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Appendix 5 Instrumentation

	Inservice Date	Serial Number
<u>GC's</u>		
Perkin-Elmer Auto System	8/92	1082103
Perkin-Elmer Auto System	8/92	2081903
Perkin-Elmer Auto System	8/92	2060611
Perkin-Elmer Auto System	8/92	610N0040401
Perkin-Elmer Auto System	8/92	2081902
Perkin-Elmer Auto System	10/96	90502
Perkin-Elmer Auto System	10/96	2081901
Hewlett Packard 5890	8/92	3203A41107
Hewlett Packard 5890	8/92	3033A3253A
Hewlett Packard 6890	12/97	4500009847
Hewlett Packard 6890	12/97	4500009866
<u>GC/MS</u>		
Hewlett Packard 5971A	8/92	3050A01784
Hewlett Packard 5971A	8/92	3188A03479
Hewlett Packard 5973	12/97	4572010693
Hewlett Packard 5973	12/97	4572010688
FTIR		
Perkin-Elmer 1625 FTIR	8/92	260F945
AA		
Perkin-Elmer 4100 ZL Furnace	8/92	6167
ICP		
Perkin-Elmer P-400 Sequential	8/92	1400030
ARL 3560 Simultaneous	12/95	5505
Leeman DRE	11/97	DR7029
Mercury Analyzer		
CETAC M 6000A Automated	3/97	049624ASX
IC		
Dionex CD20 Conductivity Detector	12/97	98010134
Dionex GP40 Gradient Pump	12/97	97120585
Dionex LC30 Chromatography Oven	12/97	98010560
Dionex AS40 Automated Sampler	12/97	98010300
TOX Analyzer		
Mitsubishi Σ_{10}	8/92	75R02913
TOC Analyzer		
Shimadzu TOC-500	8/92	28807132

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	Inservice Date	Serial Number
UV/VIS Spec		
Hitachi U1100	8/92	04059-001
Sample Introduction Devices		
Hewlett Packard Autosampler 7673A	8/92	3120A27994
OI 4560 Purge and Trap	8/92	H415460313
OI 4560 Purge and Trap	8/92	A226156
OI 4560 Purge and Trap	8/92	H415460312
OI 4560 Purge and Trap	8/92	J503460893
OI 4551 Autosampler	8/92	
OI 4551 Autosampler	8/92	
OI DPM-16 Autosampler	8/92	C420411195
OI DPM-16 Autosampler	8/92	C416411174
OI DPM-16 Autosampler	8/92	H240205
Perkin-Elmer HS-40	8/92	1413
Perkin-Elmer HS-40	8/92	1201
Perkin-Elmer HS-40	8/92	1431
Perkin-Elmer AS70	8/92	5642
Perkin-Elmer AS90	8/92	7562
CETAC ASX Autosampler	3/97	019717ASX
CETAC ASX Autosampler	1/97	091717ASX
CETAC U-5000 AT ⁺ Ultrasonic Nebulizer	4/97	049704AT+G, +E
Hewlett Packard Autosampler 6890	12/97	U574704264
Leeman	11/97	7036
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<u>Miscellaneous</u>		
Turbo Vap II Sample concentrator	8/93	46343
CEM MDS 2000 Microwave Digestion System	9/96	2216
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Data Systems Sample Master LIMS System TurboChrome Data System Hewlett Packard Chem Station/Enviroquant Dionex Peak Net 500.1