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14 March 1995

Mr. Frederick N. Mac Millan
Remedial Project Manager
U.S. Environmental Protection Agency
Hazardous Waste Management Division
Region III
841 Chestnut Street (3HW22)
Philadelphia, PA 19107



Re: Crater Resources Superfund Site

Dear Fred:

Consistent with Steve Radel's 1 March 1995 letter to you, ERM is providing the Quality Assurance Project Plan ("QAPP") Appendix A of the Crater Resources RI/FS Workplan on behalf of the Crater Resources PRP Group.

If you require additional information, please feel free to contact Steve Radel at (412) 227-2004, Gene Gonsoulin at (205) 940-7701 or me at (610) 524-3610.

Sincerely,

Richard J. Dulcey, P.E.
Project Manager

RJD/c

Enclosure-Three (3) Copies

cc: Robert Lucas, Esq.
Michele Gutman, Esq.
Douglas Schleicher, Esq.
David Armstrong, Esq.
Steven Radel
Curtis Jones, Esq.
Gene Gonsoulin, Ph.D.

AR300002

A member of the Environmental
Resources Management Group

Crater Resources PRP Committee

Quality Assurance

Project Plan

Crater Resources Site

Upper Merion Township, PA

14 March 1995

A MEMBER OF THE ENVIRONMENTAL RESOURCES MANAGEMENT GROUP

Environmental Resources Management, Inc.

855 Springdale Drive
Exton, Pennsylvania 19341



ERM.

AR300003

ERM's Commitment to Quality

Our Quality Policy

We will fully understand the requirements of our clients, our jobs, and the systems that support us.

We will conform to those requirements at all times.

Our Quality Goals

To serve you.

To serve you well.

To continually improve that service.

Our Quality Improvement Process

Train each employee.

Establish and implement requirements based on a preventative approach.

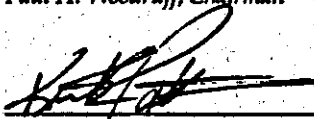
Maintain a standing Quality Improvement Team to ensure continuous improvement.

Empower Corrective Action Teams at both company-wide and local levels to correct and eliminate problems.

Continually strive to improve our client and supplier relationships.



Paul H. Woodruff, Chairman



Kent E. Patterson, President and C.E.O.

AR300004

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1.0 INTRODUCTION

This Quality Assurance Project Plan (QAPP) has been developed to present the quality assurance measures that will be used during the Remedial Investigation/Feasibility Study (RI/FS) at the Crater Resources Site, located in Upper Merion Township, Pennsylvania. The QAPP has been prepared based on guidance presented in the "Interim Guidelines and Specifications for Preparing Quality Assurance Project Plans," (QAMS-005/80, February 1983) and the "EPA NEIC Policies and Procedures Manual" (EPA Document 330/9-78-001-R, revised May 1984).

A description of the site background, site location, and previous studies is presented in Section 1.0 of the Work Plan.

2.0 PROJECT ORGANIZATION AND RESPONSIBILITIES

While all personnel involved in an investigation and in the generation of data are implicitly a part of the overall project and quality assurance program, certain individuals have specifically delegated responsibilities. Within ERM these are the Project Manager, RI Task Manager, RI Site Manager, Quality Assurance Manager, Quality Assurance Chemist, Laboratory Coordinator, and Project Geologists/Technicians. A project organization chart is presented as Figure 4-1 in the work plan. IEA Incorporated (IEA), of Whippany, New Jersey, has been identified as the project analytical laboratory. A final determination of the project analytical laboratory will be made shortly. Regardless of the final selection, the laboratory will use CLP procedures. Specific laboratory personnel with quality assurance/quality control responsibilities include the Laboratory Quality Assurance Officer and Laboratory Sample Custodian.

2.1 PROJECT MANAGER

Mr. Richard J. Dulcey is the Project Manager for the Crater Resources project. The Project Manager is an experienced manager and technical professional who provides QA review, assists in the coordination of the RI, and RA components, participates in major meetings and regulatory negotiations and provides upper level ERM contact for the client.

2.2 RI TASK MANAGER

Ms. Caroline Singler is the RI Task Manager for the Crater Resources Investigation. She is also a highly experienced manager and technical professional who will coordinate the RI on a day-to-day basis, manage all day-to-day project activities, maintain day-to-day contact with the site respondents and the USEPA, prepare project status reports, and participate in meetings and regulatory negotiations. As RI Task Manager, Ms. Singler will also be responsible for implementation and supervision of the investigation component. She will be assisted by Mr. James LaRegina, P.G., who will be the Senior Project Geologist. Mr. LaRegina will provide technical oversight and will bring a high level of experience to the more challenging aspects of this project.

2.3

RI SITE MANAGER

Mr. David Haller will be the project RI Site Manager. The RI Site Manager will be responsible for all soil boring, well installation, and sampling tasks and for the day-to-day activities of ERM field personnel. The RI Site Manager will be responsible for field quality assurance and other non-analytical data quality review. Additional responsibilities of the RI Site Manager will include the verification for accuracy of field notebooks, driller's logs, chain-of-custody records, sample labels, and other field-related documentation.

2.4

QUALITY ASSURANCE MANAGER

Ms. Shawne M. Rodgers serves as Quality Assurance Manager on all projects requiring the collection of data, and as such is not directly involved in the routine performance of technical aspects of the investigations. The Quality Assurance Manager's responsibilities include the development, evaluation, and implementation of the Quality Assurance Project Plan and procedures appropriate to the investigation. Additional responsibilities include reviewing project plans and revising the plans to ensure proper quality assurance is maintained. The Quality Assurance Manager is also responsible for all data processing activities, data processing quality control and final analytical data quality review. The Quality Assurance Manager is also responsible for final review of all Tentatively Identified Compound (TIC) mass spectra matching quality.

It is a major responsibility of the Quality Assurance Manager to ensure that all personnel have a good understanding of the project quality assurance plan, an understanding of their respective roles relative to one another, and an appreciation of the importance of the roles to the overall success of the program.

2.5

QUALITY ASSURANCE CHEMIST

Mr. Purshotam Lal Batra will serve as the Project Quality Assurance Chemist. The Quality Assurance Chemist has primary responsibility for analytical data validation and review. In this capacity, the Quality Assurance Chemist will prepare analytical quality assurance reports describing data usability and analytical quality control problems encountered.

2.6 LABORATORY COORDINATOR

Ms. Valerie Thomas serves as ERM's Laboratory Coordinator with the primary responsibilities of coordinating communication between the project team and the subcontracted laboratory. Her duties include scheduling analytical services and informing the laboratory of sample shipment and expected receipt dates; issuing ERM chain-of-custody and traffic report forms; tracking, logging, and filing documentation returned from the laboratory; and routing analytical data to the Quality Assurance Manager for validation review.

2.7 PROJECT GEOLOGISTS AND TECHNICIANS

All sampling tasks required by this investigation will be conducted by experienced environmental geologists and technicians. Their responsibilities will include the documentation of the proper sample collection protocols, sample collection and field measurements, equipment decontamination, and chain-of-custody documentation.

2.8 LABORATORY QUALITY ASSURANCE OFFICER

The volume of analytical work for a project of this size necessitates the subcontract analytical laboratory designate a Quality Assurance Officer whose duties are specific to the project. The Quality Assurance Officer has the responsibility for maintenance of all laboratory quality assurance activities in association with the project. Mr. Kevin Gorman serves as Quality Assurance Officer.

2.9 LABORATORY SAMPLE CUSTODIAN

The Sample Custodian's responsibilities include ensuring proper sample entry and sample handling procedures by laboratory personnel. Mr. John Doffinger serves as IEA's Sample Custodian.

3.0 **QUALITY ASSURANCE OBJECTIVES FOR MEASUREMENT DATA IN TERMS OF PRECISION, ACCURACY, REPRESENTATIVENESS, COMPARABILITY, AND COMPLETENESS**

3.1 **OVERALL PROJECT OBJECTIVES**

Data Quality Objectives (DQO) are quantitative and qualitative statements specifying the quality of the environmental data required to support the decision-making process. DQO define the total uncertainty in the data that is acceptable for each specific activity during the investigation. This uncertainty includes both sampling error and analytical error. Ideally, zero uncertainty is the intent; however, the variables inherently associated with the process (field and laboratory) contribute to uncertainty in the data. It is the overall project objective to keep the total uncertainty within an acceptable range that will not hinder the intended use of the data. In order to achieve this objective, data quality requirements such as quantitation limits, criteria for accuracy and precision, sample representativeness, data comparability, and data completeness have been specified. The overall data quality objectives and requirements will be established such that there is a high degree of confidence in measurements performed during the project.

Data collection requirements are included in this QAPP. Data Quality Objectives for the RI activities are discussed below.

Data Quality Objectives (DQOs) are quantitative and qualitative statements that are specified to ensure that data of known and appropriate quality are obtained to support their intended use, i.e., the risk assessment and selection of an appropriate remedy for the Site. The DQOs with respect to the field investigation are to maximize confidence in the data with respect to precision, accuracy, representativeness, completeness, and comparability. The DQOs identified for this investigation include:

1. Are constituents of interest (COIs) present or absent in the contents of the three former quarries or in the soils immediately adjacent to Quarry No. 3 along the former pipeline?
2. If COIs are present in the former quarries, what quantities remain in the surficial and subsurface materials?
3. What impact, if any, have Quarries No. 1, No. 2 and No. 3 had on ground water?

4. What are the characteristics of the ground water migration pathways?
5. What are the risks, if any, to human health and the environment associated with the site?
6. What degree of COI reduction, if any, will mitigate potential ground water impacts?

Subsurface soil, surficial soil, surface water, sediment, and ground water samples will be collected and submitted to the laboratory for chemical and physical analysis.

As stated earlier, the parameters that will be used to specify data quality requirements and to evaluate the analytical system performance are precision, accuracy, representativeness, completeness, and comparability (PARCC). Table 3-1 presents definitions for these parameters.

Table 3-1 *Definitions of Data Quality Parameters*

Precision	A measure of the reproducibility of measurements under a given set of conditions.
Accuracy	A measure of the bias that exists in a measurement system.
Representativeness	The degree to which sample data accurately and precisely represent selected characteristics.
Completeness	A measure of the amount of valid data obtained from the measurement system compared to the amount that is required.
Comparability	A measure of confidence with which one data set can be compared with another.

3.2

FIELD INVESTIGATION QUALITY OBJECTIVE

The objective with respect to the field investigation is to maximize the confidence in the data in terms of PARCC.

Section 9.0 presents the frequency with which trip blanks, field duplicates, equipment blanks, and matrix spikes will be collected such that a specific degree of precision and accuracy can be calculated. The data quality objective for field duplicates is to achieve precision equal to or greater than that summarized on Table 3-2.

Precision will be calculated as the relative percent difference (RPD) if there are only two analytical points and as relative standard deviation (RSD) if there are more than two analytical points. The submission of trip blanks and field blanks will provide a check on accuracy. Although accuracy is best assessed by evaluating the results of blanks, blanks do not monitor analyte losses. The submission of blanks will, however, monitor contaminants introduced with the sampling process, preservation, handling, shipping, and the analytical process. The data quality objective for trip blanks and field blanks is to meet or exceed the contract required quantitation limit (CRQL, CLP analyses) or the quantitation limit (non-CLP parameters). In the event that the blanks are contaminated and/or poor field duplicate precision is obtained, the associated data will be qualified as described in Section 12.3. Through the submission of field QC samples, the distinction can be made between laboratory problems, sampling technique, and sample matrix variability.

Table 3-2 Criteria Objectives

Precision Objectives	Aqueous	Solid/Other
Field Duplicate/Replicates (Blind or labeled)		
TCL VOA	within 20% RPD	within 30% RPD
TCL BNA	within 25% RPD	within 40% RPD
TAL Inorganics	within 25% RPD	within 40% RPD
Misc. Parameters	within 25% RPD	within 40% RPD
Laboratory Duplicates (Unspiked)		
TAL Inorganics	As specified in current CLP SOW	As specified in current CLP SOW
Laboratory Duplicate (MSD)		
TCL VOA	As specified in current CLP SOW	As specified in current CLP SOW
TCL BNA	As specified in current CLP SOW	As specified in current CLP SOW
Accuracy Objectives		
Field or trip blanks		
TCL VOA	Less than the CRQL	Less than the CRQL
TCL BNA	Less than the CRQL	Less than the CRQL
TAL Inorganics	Less than the CRDL	Less than the CRDL
Misc. Parameters	Less than the QL	Less than the QL
Laboratory Blanks		
TCL VOA	As specified in current CLP SOW	As specified in current CLP SOW
TCL BNA	As specified in current CLP SOW	As specified in current CLP SOW
TAL Inorganics	As specified in current CLP SOW	As specified in current CLP SOW
Misc. Parameters	Less than the QL	Less than the QL
Matrix spikes/surrogate spikes		
All TCL Fractions	As specified in current CLP SOW	As specified in current CLP SOW
Post - digestion spikes	As specified in current CLP SOW	As specified in current CLP SOW
TAL Inorganics	As specified in current CLP SOW	As specified in current CLP SOW
Misc. Parameters	As specified in Attachment 1	As specified in Attachment 1

NA - Non Applicable.

Precision and accuracy for the field pH and conductivity measurements are dependent on the type and condition of the instrument used and the care used in the standardization and operation. The precision and accuracy objectives for the instrumentation used are as follows:

- pH precision will be ± 0.3 pH standard units and an accuracy of ± 0.03 pH standard units. pH measurements will be reported to two significant figures.
- Conductivity precision will be ± 3 $\mu\text{mhos/cm}$ on the 500 $\mu\text{mhos/cm}$ range, ± 25 $\mu\text{mhos/cm}$ on the 5,000 $\mu\text{mhos/cm}$ range, and ± 250 $\mu\text{mhos/cm}$ on the 50,000 $\mu\text{mhos/cm}$ range. Accuracy for the conductivity measurements is a function of the conductivity reading for the probe and instrument combined. Conductivity measurements will be reported to one significant figure for values below ten and to two significant figures for values above ten.

To ensure sample representativeness, sample collection will be performed in strict accordance with EPA-recommended procedures for collection and preservation including EPA-recommended holding times specified in EPA 600/4-79-020 (Methods for Chemical Analysis of Water and Wastes), the Federal Register, 26 October 1984, and the specific Contract Laboratory Program (CLP) Statements of Work (SOW) the Analytical Laboratory is operating under.

The data quality objective for the completeness of data with respect to the sampling (field investigation) is 100%. Although this goal appears rather ambitious, it can be attained. In the event 100% is not obtained, the effect of the uncollected data will be evaluated by the Project Manager as to its impact (if any) on project objectives. Corrective actions will be initiated to resolve any data gaps from the original objectives, found as a result of less than 100% data completeness. Every effort will be made to obtain valid data for all sampling points, particularly those considered to be critical points. In this regard, the critical point samples which are identified will necessarily be selected as subsequent QC samples (duplicate and matrix spikes) at the frequency specified in Section 9.0.

In order to establish a degree of comparability such that observations and conclusions can be directly compared with all historical data, ERM will use standardized methods of field analysis, sample collection, holding times, and preservation. In addition, field conditions will be considered in evaluating sampling results in order to attain a high degree of data comparability.

3.3

LABORATORY DATA QUALITY OBJECTIVES

The laboratory will demonstrate analytical precision and accuracy by the analysis of laboratory duplicates and matrix spike duplicates. Precision (as well as instrument stability) will also be demonstrated by comparison of response factors for calibration standards. Laboratory accuracy will be demonstrated by the addition of surrogate and matrix spike compounds. Accuracy will be presented as percent recovery (R). Precision will be presented as relative percent differences (RPD), relative standard deviation (RSD), or percent difference (PD), whichever is applicable to the type of QC samples involved. Laboratory method blanks will also demonstrate accuracy with respect to the analyses. The frequencies of laboratory duplicates, matrix spikes and laboratory blanks are specified in Section 9.0. As considerable reference is made to Attachment 1 in the remainder of this QAPP, it is suggested it be reviewed at this time. IEA's data quality objectives are detailed in Attachment 1.

The analysis laboratory will be expected to process (purge, extract, or digest) an aliquot of sample such that the analytical results will provide a high degree of representation with respect to the sampling point. In addition, the analysis laboratory will be expected to document all analytical problems encountered during the course of the investigation. Communication will be maintained with the laboratory so that analytical problems encountered with critical sample points will allow these samples to be re-collected, if necessary. Further, the laboratory will be required to provide all data packages in the CLP deliverable format (most recent) or CLP-equivalent to ensure that analytical methods, parameters, and reporting units are compatible throughout the investigation.

3.4

CRITERIA OBJECTIVES

The quantitative objectives (criteria) that ERM will require for both field and laboratory accuracy and precision are summarized in Table 3-2.

The laboratory will be expected (as an ideal objective) to report the contract required quantitation/detection limits (CRQL/CRDL) for all samples in the appropriate statistical reporting units for all analyses, as stated in the CLP SOWs. However, it should be noted that actual quantitation limits are sample specific and depend on variables such as dilution factors, sample matrices, percent moisture, and the specific analyte. The data reported at or near the CRQL/CRDL will be handled cautiously since the stated data quality objectives for accuracy and

precision may not "translate" well in some situations (i.e., accuracy and precision suffer for results near the CRQL/CRDL).

3.5

DATA MANAGEMENT OBJECTIVES

It is a data management objective that all aspects of the investigation from sample design, collection, shipment, analysis use/decisions, etc. be performed in conjunction with rigorous QA/QC documentation. The specific details of this documentation can be found throughout this document.

It is expected that by the design of separate data quality requirements for field sampling and laboratory analysis, clear distinctions can be made such that any problems found in the system can be isolated with respect to the cause. Conversely, the data quality requirements are also designed to provide an indication of the variability inherent to the overall system.

The overall data management objective is to provide a complete data base with a high degree of confidence through the use of a phased approach of sampling, analysis, data assessment (data review), data qualification, and feedback.

4.0 SAMPLING PROCEDURES

The numbers of samples, locations, and justifications for each sample media to be collected are presented in the Crater Resources Work Plan. Please refer to the Work Plan for this information. Procedures associated with the subsurface soil, surficial soil, surface water, sediment and ground water sampling are described in Sections 3 and 4 of the Sampling and Analysis Plan. Table 4-1 presents a Sample Summary Matrix for the Crater Resources sampling activities.

4.1 DECONTAMINATION AND POST-SAMPLING PROCEDURES

Decontamination of equipment will take place at a specific decontamination zone designed at the site.

4.1.1 Evacuation and Drilling Equipment Decontamination

Evacuation and drilling equipment will be decontaminated prior to initial use, between boring locations, and at the completion of drilling activities. Items necessary to decontaminate include:

- back of drilling rig
- auger flights
- down-hole equipment
- backhoe bucket

A manual scrubbing to remove foreign material followed by a thorough steam cleaning will be used for decontamination of the above items. Drilling equipment will be stored in a contaminant-free location above ground on wooden supports after decontamination and covered with plastic until use.

4.1.2 Sampling Equipment Decontamination

All non-disposable equipment (hand augers, split spoon samplers, hand trowels, etc.) will be decontaminated according to the procedures summarized below:

- Manual scrub with non-phosphate soap solution plus tap water wash
- Tap water rinse

- 10% nitric acid rinse (for metals only)
- Pesticide grade methanol rinse (for organics only)
- Distilled/Deionized water rinse
- Air dry

4.1.3

Sample Preparation and Preservation

Immediately after collection, samples will be transferred to properly labeled (see Section 5.0 of this QAPP) sample containers with all necessary preservatives added. Table 4-1 lists the proper container material, volume requirement, and preservation needed for the Crater Resources investigation. Samples requiring refrigeration for preservation will be immediately transferred to coolers packed with ice or ice packs. Proper chain-of-custody documentation will be maintained as discussed in Section 5.0 of this QAPP.

Table 4-1
Soil Sample Matrix Summary
Remedial Investigation
Coker Resource Site
Upper Merion Township, Pennsylvania

Area	Number of Samples*	Number of Trip Blanks**	Number of Ambient (Field) Blanks	Number of Equipment (Field) Blanks	Number of Duplicates	Number of Matrix Spikes	Number of Matrix Spikes Duplicates***	Total Number of Samples	Analytes	Analytical Method	Container and Preservation	Analyte Holding Time****
Quarry No. 3 (Four Surface and Four Subsurface Soil Samples)	0	1	1	1	1	1	1	14	TCL Volatiles	QLM02	240 ml clear glass vials w/ Teflon lined enclosure. Cool to 4° C.	14 days
	0	0	1	1	1	1	1	13	TCL Semivolatiles	QLM02	1-liter amber glass w/ Teflon lined enclosure. Cool to 4° C.	7 days if extractors; analysis w/ 40 days of extract preparation.
	0	0	1	1	1	1	1	13	PCBs	B080	Same Container as above.	7 days if extractors; analysis w/ 40 days of extract preparation.
	0	0	1	1	1	1	1	13	TAL Metals	LM02	Same Container as above.	180 days; 28 days for mercury.
	0	0	1	1	1	1	1	13	TAL Cyanide	LM02	Same Container as above.	14 days
	0	0	1	1	1	1	1	13	Sulfate	9038.9 (modified)	Same Container as above.	28 days
	0	0	1	1	1	1	1	13	Ammonia-Nitrogen	350.3 (modified)	Same Container as above.	28 days
	0	0	1	1	1	1	1	13				

* The number of samples may change based on decisions made in the field.
 ** Trip blanks will be submitted for volatile analysis at a frequency of 1 per cooler per day. The number of trip blanks may change based on time spent in the field.
 *** Samples will be collected at a frequency of 1 per 20 samples for MS/MSD analysis. Inorganic analysis will include a single matrix spike and a laboratory duplicate versus a matrix spike duplicate.
 **** Holding times are from the date of sample collection.

Area	Number of Samples*	Number of Trip Blank**	Number of Ambient (Field) Blank	Number of Equipment (Field) Blank	Number of Duplicates	Number of Matrix Spikes	Number of Matrix Spikes Duplicates***	Total Number of Samples	Analytes	Analytical Method	Container and Preservation	Analysis Holding Time****
Quarry No. 3 Paved Area Sediments	3	1	1	1	1	1	1	8	TCL Volatiles	CLM02	2-40 ml clear glass vials w/ Teflon lined enclosure. Cool to 4° C.	14 days
	3	0	1	1	1	1	1	8	TCL Semivolatiles	CLM02	1-Liter amber glass w/ Teflon lined enclosure. Cool to 4° C.	7 days if extracted; analysis w/ 40 days of extract preparation.
	3	0	1	1	1	1	1	8	PCBs	8090	Same Container as above.	7 days if extracted; analysis w/ 40 days of extract preparation.
	3	0	1	1	1	1	1	8	TAL Metals	LM02	Same Container as above.	140 days; 28 days for mercury.
	3	0	1	1	1	1	1	8	TAL Cyanide	LM02	Same Container as above.	14 days
	3	0	1	1	1	1	1	8	Sulfide	9030.0 (modified)	Same Container as above.	28 days
	3	0	1	1	1	1	1	8	Ammonia-Nitrogen	350.3 (modified)	Same Container as above.	28 days
	3	0	1	1	1	1	1	8	Chemical Oxygen Demand (COD)	410.1	Same Container as above.	28 days
	3	0	1	1	1	1	1	8	Total Organic Carbon	415.1	Same Container as above.	28 days

* The number of samples may change based on decisions made in the field.
 ** Trip blanks will be submitted for volatile analysis at a frequency of 1 per cooler per day. The number of trip blanks may change based on time spent in the field.
 *** Samples will be collected at a frequency of 1 per 20 samples for MS/MSD analysis. Inorganic analysis will include a single matrix spike and a laboratory duplicate versus a matrix spike duplicate.
 **** Holding times are from the date of sample collection.

Table 4-1
Soil Sample Matrix Summary
Nonradial Investigation
Coker Resources Site
Upper Merion Township, Pennsylvania

Area	Number of Samples*	Number of Trip Blank/Lot**	Number of Ambient (Field) Blanks	Number of Equipment (Field) Blanks	Number of Duplicates	Number of Matrix Spikes	Number of Matrix Spikes Duplicated***	Total Number of Samples	Analyte	Analytical Method	Container and Preservation	Analysis Holding Time****
Quarry No. 1 and 2 (Six Surficial and Six Subsurface Soil Samples)	12	2	1	1	1	1	1	19	TCL Volatiles	GLMC2	2-40 ml clear glass vials w/ Teflon lined enclosure. Cool to 4° C.	14 days
	12	0	1	1	1	1	1	17	TCL Semivolatiles	GLMC2	1-liter amber glass w/ Teflon lined enclosure. Cool to 4° C.	14 days @ extraction; analysis w/ 40 days of extract preparation.
	6	0	1	1	1	1	1	11	PCBs	GLMC2	Same Container as above.	14 days @ extraction; analysis w/ 40 days of extract preparation.
	12	0	1	1	1	1	1	17	TAL Metals	ELM2	Same Container as above.	180 days; 28 days for mercury.
	12	0	1	1	1	1	1	17	TAL Cyanide	ELM2	Same Container as above.	14 days
	12	0	1	1	1	1	1	17	Sulfide	9039.0 (modified)	Same Container as above.	28 days
	12	0	1	1	1	1	1	17	Ammonia-Nitrogen	350.3 (modified)	Same Container as above.	28 days
	6	0	1	1	1	1	1	11	PCBs	9080	Same Container as above.	7 days @ extraction; analysis w/ 40 days of extract preparation.
Pond/Dewatered Background Soils	6	2	1	1	1	1	1	13	TCL Volatiles	GLMC2	2-40 ml clear glass vials w/ Teflon lined enclosure. Cool to 4° C.	14 days
	6	0	1	1	1	1	1	11	TCL Semivolatiles	GLMC2	1-liter amber glass w/ Teflon lined enclosure. Cool to 4° C.	14 days @ extraction; analysis w/ 40 days of extract preparation.
	6	0	1	1	1	1	1	11	PCBs	9080	Same Container as above.	7 days @ extraction; analysis w/ 40 days of extract preparation.
	6	0	1	1	1	1	1	11	TAL Metals	ELM2	Same Container as above.	180 days; 28 days for mercury.
	6	0	1	1	1	1	1	11	TAL Cyanide	ELM2	Same Container as above.	14 days
	6	0	1	1	1	1	1	11	Sulfide	9039.0 (modified)	Same Container as above.	28 days
	6	0	1	1	1	1	1	11	Ammonia-Nitrogen	350.3 (modified)	Same Container as above.	28 days
	6	0	1	1	1	1	1	11	PCBs	9080	Same Container as above.	7 days @ extraction; analysis w/ 40 days of extract preparation.

* The number of samples may change based on decisions made in the field.
 ** Trip blanks will be submitted for volatile analysis at a frequency of 1 per cooler per day. The number of trip blanks may change based on time spent in the field.
 *** Samples will be collected at a frequency of 1 per 20 samples for MS/MSD analysis. Inorganic analysis will include a single matrix spike and a laboratory duplicate versus a matrix spike duplicate.
 **** Holding times are from the date of sample collection.
 ***** Analysis of these samples is contingent on detection of PCBs in Quarry 2 & 3 solid samples.

Section 4
Water Sample Matrix Summary
Upper Merion Township, Pennsylvania

Area	Number of Samples*	Number of Trip Blank**	Number of Ambient (Field) Samples	Number of Equipment (Field) Samples	Number of Duplicate	Number of Matrix Spikes	Number of Matrix Spikes Duplicate***	Total Number of Samples	Analyte	Analytical Method	Container and Preservation	Analyte Holding Time****
Quarry No. 3 Ponded Area Surface Water/Liquid Below Paved Sidewalk	5	1	1	1	1	1	1	11	TOL Volatiles	OLM02	2-40 ml clear glass vials w/ Teflon lined enclosure. HCl to pH 5.2. Cool to 4° C.	14 days
	3	0	1	1	1	1	1	8	TOL Semivolatiles	OLM02	2-liter amber glass w/ Teflon lined enclosure. Cool to 4° C.	7 days in extraction; analyze w/ 40 days of extract preparation.
	3	0	1	1	1	1	1	8	PCBs****	8090	2-liter amber glass w/ Teflon lined enclosure. Cool to 4° C.	7 days in extraction; analyze w/ 40 days of extract preparation.
	3	0	1	1	1	1	1	8	TAL Metals	ELM02	1-liter plastic. HNO3 to pH 5.2. Cool to 4° C.	100 days; 28 days for mercury.
	3	0	1	1	1	1	1	8	TAL Metals (dissolved)	ELM02	1-liter plastic; acid blanked. HNO3 to pH 5.2. Cool to 4° C.	100 days; 28 days for mercury.
	3	0	1	1	1	1	1	8	TAL Cyanide	ELM02	1-liter glass w/ Teflon lined enclosure. NaOH to pH 12. Cool to 4° C.	14 days
	3	0	1	1	1	1	1	8	Total Suspended Solids	160.2	1-liter plastic. Cool to 4° C.	7 days
	3	0	1	1	1	1	1	8	Total Dissolved Solids	580.1	Same Container as above.	7 days
	3	0	1	1	1	1	1	8	Alkalinity (Total Carbonate, Bicarbonate)	310.1	1-liter plastic. Cool to 4° C.	48 hours
	3	0	1	1	1	1	1	8	Sulfate	9038.0 (modified)	Same Container as above.	28 days
	3	0	1	1	1	1	1	8	Ammonia-Nitrogen	350.3 (modified)	1-liter plastic. H2SO4 to pH 5.2. Cool to 4° C.	28 days
	3	0	1	1	1	1	1	8	Biochemical Oxygen Demand (BOD)	405.1	1-liter plastic. Cool to 4° C.	48 hours
	3	0	1	1	1	1	1	8	Chemical Oxygen Demand (COD)	410.1	1-liter plastic. H2SO4 to pH 5.2. Cool to 4° C.	28 days
	3	0	1	1	1	1	1	8	Mercury	130.2	Same Container as TAL metals. Cool to 4° C.	100 days
	3	0	1	1	1	1	1	8	Total Organic Carbon	415.1	1-liter plastic. H2SO4 to pH 5.2. Cool to 4° C.	28 days

* The number of samples may change based on decisions made in the field.
 ** Trip blanks will be submitted for volatile analysis at a frequency of 1 per cooler per day. The number of trip blanks may change based on time spent in the field.
 *** Samples will be collected at a frequency of 1 per 20 samples for MS/MSD analysis. Inorganic analysis will include a single matrix spike and a laboratory duplicate versus a matrix spike duplicate.
 **** Holding times are from the date of sample collection.
 ***** Analysis of these samples is contingent on detection of PCBs in Quarry 2 & 3 solid samples.

Table 4-1
Soil Sample Matrix Summary
Remedial Investigation
Crater Remediation Site
Upper Merion Township, Pennsylvania

Area	Number of Samples	Number of Trip Blanks	Number of Ambient (Field) Blanks	Number of Equipment (Field) Blanks	Number of Duplicate Samples	Number of Matrix Spikes Duplicate	Total Number of Samples	Analytes	Analytical Method	Container and Preservation	Analyte Holding Time
Ground Water Near Monitoring Wells	14	2	0	0	1	1	19	TCL Volatiles	GLM02	240 ml clear glass vials w/ Teflon lined enclosure. HCl to pH 12. Cool to 4° C.	14 days
	14	0	1	1	1	1	19	TCL Semivolatiles	GLM02	2-liter amber glass w/ Teflon lined enclosure. Cool to 4° C.	7 days in extraction; analysis w/ 40 days of extract preparation.
	14	0	1	1	1	1	19	PCBs****	8080	2-liter amber glass w/ Teflon lined enclosure. Cool to 4° C.	7 days in extraction; analysis w/ 40 days of extract preparation.
	14	0	1	1	1	1	19	TAL Metals	ELM02	1-liter plastic. HNO3 to pH 12. Cool to 4° C.	180 days; 28 days for mercury.
	14	0	1	1	1	1	19	TAL Metals (dissolved)	ELM02	1-liter plastic. HNO3 to pH 12. Cool to 4° C.	180 days; 28 days for mercury.
	14	0	1	1	1	1	19	TAL Cyanide	ELM02	1-liter glass w/ Teflon lined enclosure. NaOH to pH 12. Cool to 4° C.	14 days
	14	0	1	1	1	1	19	Sulfate	375.4	1-liter plastic. Cool to 4° C.	28 days
	14	0	1	1	1	1	19	Ammonia-Nitrogen	350.3	1-liter plastic. H2SO4 to pH 12. Cool to 4° C.	28 days

* The number of samples may change based on decisions made in the field.
 ** Trip blanks will be submitted for volatile analysis at a frequency of 1 per cooler per day. The number of trip blanks may change based on time spent in the field.
 *** Samples will be collected at a frequency of 1 per 20 samples for MSMSD analysis. Inorganic analysis will include a single matrix spike and a laboratory duplicate versus a matrix spike duplicate.
 **** Holding times are from the date of sample collection.
 ***** Analysis of these samples is contingent on detection of PCBs in Quarry 2 & 3 solid samples.

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Area	Number of Samples*	Number of Trip Blanks**	Number of Ambient (Field) Blanks	Number of Equipment (Field) Blanks	Number of Duplicates	Number of Matrix Spikes Duplicates***	Total Number of Samples	Analytes	Analytical Method	Container and Preservation	Analyte Holding Time****
Ground Water Existing Monitoring Wells (or Replacement Wells)	3	1	1	1	1	1	9	TCL Volatiles	GLM02	2-40 ml clear glass vials w/ Teflon lined enclosures. HCl to pH <2. Cool to 4° C.	14 days
	3	0	1	1	1	1	8	TCL Semivolatiles	GLM02	2-liter amber glass w/ Teflon lined enclosures. Cool to 4° C.	7 days in extraction; analysis within 40 days of extract preparation.
	3	0	1	1	1	1	8	PCBs****	9090	2-liter amber glass w/ Teflon lined enclosures. Cool to 4° C.	7 days in extraction; analysis within 40 days of extract preparation.
	3	0	1	1	1	1	8	T/M Metals	LM02	1-liter plastic. HNO ₃ to pH <2. Cool to 4° C.	180 days; 28 days for recovery.
	3	0	1	1	1	1	8	T/M Metals (Measured)	LM02	1-liter plastic field stored. HNO ₃ to pH <2. Cool to 4° C.	180 days; 28 days for recovery.
	3	0	1	1	1	1	8	T/M Cyanide	LM02	1-liter glass w/ Teflon lined enclosures, NaOH to pH <12. Cool to 4° C.	14 days
	3	0	0	0	0	0	3	Sulfide	275.4	1-liter plastic. Cool to 4° C.	28 days
	3	0	0	0	0	0	3	Ammonia-Nitrogen	350.3	1-liter plastic. H ₂ SO ₄ to pH <2. Cool to 4° C.	28 days

* The number of samples may change based on decisions made in the field.

** Trip blanks will be submitted for volatile analysis at a frequency of 1 per cooler per day. The number of trip blanks may change based on time spent in the field.

*** Samples will be collected at a frequency of 1 per 20 samples for MS/MSD analysis. Inorganic analysis will include a single matrix spike and a laboratory duplicate versus a matrix spike duplicate.

**** Holding times are from the date of sample collection.

***** Analysis of these samples is contingent on detection of PCBs in Quarry 2 & 3 solid samples.

Table 4-1
Ground Water Sample Matrix Summary
Remedial Investigation
Coker Resource Site
Upper Merion Township, Pennsylvania

Area	Number of Samples*	Number of Trip Blanks**	Number of Ambient (Field) Blanks	Number of Equipment (Field) Blanks	Number of Duplicates	Number of Matrix Spikes	Number of Matrix Spikes Duplicate***	Total Number of Samples	Analyte	Analytical Method	Container and Preservation	Analyte Holding Time***
Ground Water Oil Site Wells	2	1	1	1	1	1	1	8	TCL Volatiles	CLM02	2-40 ml clear glass vials w/ Teflon lined enclosures, HCl to pH <2. Cool to 4° C.	14 days
	2	0	1	1	1	1	1	7	12	CLM02	2-liter amber glass w/ Teflon lined enclosures, Cool to 4° C.	7 days in extraction; analysis w/ 40 days of extract preparation.
	2	0	1	1	1	1	1	7	PCBs****	8080	2-liter amber glass w/ Teflon lined enclosures, Cool to 4° C.	7 days in extraction; analysis w/ 40 days of extract preparation.
	2	0	1	1	1	1	1	7	TAL Metals	LM02	1-liter plastic, HNO3 to pH <2, Cool to 4° C.	180 days; 28 days for mercury.
	2	0	1	1	1	1	1	7	TAL Metals (dissolved)	LM02	1-liter plastic, field filtered, HNO3 to pH <2, Cool to 4° C.	180 days; 28 days for mercury.
	2	0	1	1	1	1	1	7	TAL Cyanide	LM02	1-liter glass w/ Teflon lined enclosures, NaOH to pH <12, Cool to 4° C.	14 days
	2	0	1	1	1	1	1	7	Sulfate	375.4	1-liter plastic, Cool to 4° C.	28 days
	2	0	1	1	1	1	1	7	Ammonia-Nitrogen	350.3	1-liter plastic, H2SO4 to pH <2, Cool to 4° C.	28 days

* The number of samples may change based on decisions made in the field.
 ** Trip blanks will be submitted for volatile analysis at a frequency of 1 per cooler per day. The number of trip blanks may change based on time spent in the field.
 *** Samples will be collected at a frequency of 1 per 20 samples for MS/MSD analysis. Inorganic analysis will include a single matrix spike and a laboratory duplicate versus a matrix spike duplicate.
 **** Holding times are from the date of sample collection.
 ***** Analysis of these samples is contingent on detection of PCBs in Quarry 2 & 3 solid samples.

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5.0 SAMPLE CUSTODY

The primary objective of sample custody procedures is to create an accurate written record which can be used to trace the possession and handling of all samples from the moment of their collection, through analysis, until their final disposition. Custody for samples collected during this investigation will be maintained by the RI Site Manager or the field personnel collecting the samples. The RI Site Manager or the field personnel will be responsible for documenting each sample transfer and maintaining custody of all samples until they are shipped to the laboratory.

ERM will use laboratory-supplied I-Chem 200 Series or equivalent bottles appropriate for each media as sample containers. All necessary chemical preservatives will be added to the bottles by the laboratory prior to the sampling event, where appropriate. Preservative type and source will be documented in ERM's preservation log. Custody of the sample bottles will be maintained by the RI Site Manager. Sample bottles needed for a specific sampling task will then be relinquished by the RI Site Manager to the sampling team after the RI Site Manager has verified the integrity of the bottles and ensured that the proper bottles have been assigned to the task to be conducted.

A self-adhesive sample label will be affixed to each container before sample collection. At a minimum, the sample label will contain the following information, as shown on Figure 5-1.

- Client - Job Name (Crater Resources)
- ERM Traffic Report Number
- Sample identification - place of sampling
- Date and time collected
- Sampler's initials
- Testing required
- Preservatives added

Immediately after sample collection, each sample bottle will be sealed in an individual plastic bag. Samples will then be placed immediately into an insulated cooler for shipment to the laboratory. ERM field Chain-of-Custody records (Figure 5-2) and an ERM Traffic Report (Figure 5-3)

Figure 5-1
Sample Container Labels
Crater Resources PRP Group

Client/W.O.#:		Traffic Report #:	
Sample Identification:			
Collection Information:		☐ Composite	☐ Grab
Date:	Time:	By:	
Testing Required:			
Preservative:			
Environmental Resources Management, Inc. 855 Springdale Dr. • Exton, Pennsylvania 19341 • (215) 524-3500			

Client/W.O. #		
Traffic Report/Sample I.D.		
Collection Information:		
Date:	By:	Time:
Preservatives:		
Environmental Resources Management, Inc. • Exton, Pennsylvania 19341 • (215) 524-3500		

Figure 5-2

[illegible]

Figure 5-3
ERM Traffic Report Form
Crater Resources PRP Group

Traffic Report			
1 Project W.O.		2 Sample Concentration	
Project Name/Location		☐ Low Concentration ☐ Medium Concentration	
		3 Ship to:	
		5 Sampling Personnel Contact	
4 Sample Matrix		Sampler	
☐ Liquid ☐ Solid		Project Manager	
☐ Other		Phone No. (215) 524-3500	Attn.:
6 Shipping Information		7 Specify Type of Analyses, Number of Containers, Approx. Volume	
(Name of Carrier)		Analyses/Method Requested	No. of Bottles
(Date Shipped)			Total Volume
(Airbill Number)			
8 Sample Location			
Date:			
Time:			
9 Sample Description		10 Special Handling (e.g. Safety Procedures/Hazardous)	
☐ Surface Water	☐ Soil		
☐ Ground Water	☐ Solid		
☐ Leachate	☐ Other:	Additional Comments (Specify date package, rush work, special detection limits, etc.)	
☐ Sediment			
11 Condition of Samples Received (to be completed by Laboratory Log-In.)			
☐ Samples received intact			
☐ Samples at 4 degrees (C)		Log-In Person's Signature	
☐ Samples not leaking			
☐ Container numbers match as specified in Item 7			
☐ Container tags match Chain of Custody			
☐ Cooler received with Custody Seals intact		☐ Samples contained within plastic bags	

completed at the time of sample collection will accompany the samples inside the cooler for shipment to the laboratory. The samples will be properly relinquished on the field Chain-of-Custody record by the sampling team. These record forms will be sealed in a ziploc plastic bag to protect them against moisture. Each cooler will contain sufficient ice and/or ice packs to ensure that proper temperature is maintained and will be packed in a manner to prevent damage to sample containers. The RI Site Manager will then initial and custody seal (Figure 5-4) each sample cooler. All coolers will be shipped by an overnight courier according to current US DOT regulations. Prior to releasing the coolers, the RI Site Manager will require the courier to sign an ERM Cooler Transfer Acknowledgment (Figure 5-5). Upon receiving the samples, the laboratory Sample Custodian will inspect the condition of the samples, compare the information on the sample labels against the field Chain-of-Custody record and Traffic Reports, assign a IEA control identification number, and log the control number into the computer sample inventory system.

The preparation of all sample bottles (cleaning technique, preservative added, etc.) will be documented. When samples requiring preservation by either acid or base are received at the laboratory, the pH will be measured and documented. The Laboratory Sample Custodian will then store the sample in a secure sample storage cooler maintained at 4°C and maintain custody until the sample is assigned to an analyst for analysis. Custody will be maintained until disposal of the analyzed samples.

The Laboratory Sample Custodian will note any damaged sample containers or discrepancies between the sample label and information on the field Chain-of-Custody record logging the sample and will note any discrepancies in Section 11.0 of the ERM Traffic Report. This information will also be communicated to the RI Site Manager or field personnel so that proper action can be taken. The Chain-of-Custody form will be signed by both the relinquishing and receiving parties each time the sample changes hands, and the reason for transfer indicated.

Figure 5-4
ERM Custody Seal
Crater Resources PRP Group

 ERM	OFFICIAL CUSTODY SEAL	Name: _____
		Date: _____

Figure 5-5
ERM Cooler Transfer Acknowledgement
Crater Resources PRP Group

[illegible]

6.0 CALIBRATION PROCEDURES AND FREQUENCY

6.1 LABORATORY CALIBRATION

Laboratory calibration and frequency is specified in the Contract Laboratory Program (CLP) Statement of Work (SOW) methods for both the TCL organics and TAL inorganics and is summarized in Attachment 1. Laboratory calibration and frequency for non-CLP analyses is also discussed in Attachment 1.

6.2 FIELD CALIBRATION

In addition to the laboratory analyses conducted during the course of this investigation, field measurements of pH, Eh, specific conductance, and temperature will be taken for the ground water and surface water samples will be performed. Project geologists and field technicians will be responsible for the calibration of field equipment. Calibration will be recorded directly into the field notebook.

Field equipment will be calibrated using standard solutions which have certified concentrations. These standards will be purchased from chemical supply houses. The frequency of field calibration procedures will, at a minimum, include the following:

- The specific conductance and pH meter will be calibrated a minimum of once at the beginning of the day and documented in the calibrator's field book (see Section 8.0). Calibration will be checked mid-day and at the end of the day to ensure proper measurements are taken.
- pH and Eh meters will be calibrated using specific techniques according to the manufacturer's instructions (Attachment 3) and two standard buffer solutions (either pH 4 and 7, or 7 and 10) obtained from chemical supply houses. The pH value of these buffers will be compensated for temperature according to the values supplied on the manufacturer's bottle label. The temperature (measured as below) at which the sample pH was measured will then be used to compensate for temperature on the meter. The same standard buffer solutions will be used to check the pH meter calibration.

- The DO meter will be calibrated as specified in Attachment 4.
- Temperature measurements will be performed using a field thermometer (Thomas Scientific Company No. 9329A10, or equivalent) and recorded to ± 0.2 degrees Celsius. The thermometer will be calibrated once to a certified NBS thermometer. Temperature measurements will be taken with the field thermometer in ice water and boiling water, and compared to those measured with the certified NBS thermometer. The appropriate calibration factor for the field thermometer will be recorded and marked on the thermometer case.
- Specific conductance meters will be calibrated according to manufacturer's instructions (Attachment 4) using a 1413.0 μmhos (KCI) solution prepared by ERM. The conductivity probe cell constant will be calculated according to the formula:

$$K = \frac{1413.0 (C)^1}{1 + 0.02 (T - 25^\circ\text{C})}$$

Where:

K = probe cell constant (unitless)
C = measured conductance value of standard
T = temperature ($^\circ\text{C}$) of standard

Table 6-1 will be used to correct for the standard solution's conductivity value if it is not at 25°C .

Table 6-1 Conductivity Temperature Corrections for 1,413 $\mu\text{MHOS/CM}$ Conductivity Standard

Temperature $^\circ\text{C}$	$\mu\text{mhos/cm}$
15	1,141.5
16	1,167.5
17	1,193.5
18	1,219.9
19	1,246.4
20	1,273.0
21	1,299.7
22	1,326.6
23	1,353.6
24	1,380.8
25	1,408.1
26	1,436.5
27	1,463.2
28	1,490.9
29	1,518.7
30	1,546.7

Using the cell constant calculated above and the following formula, field specific conductance measurements will be corrected to 25°C.

$$S = \frac{K \cdot C}{(1 + 0.02 (T - 25))}$$

Where:

S = Specific conductance at 25°C (µmhos/cm)
K = calculated cell constant
C = field specific conductance (µmhos/cm)
T = temperature (°C) of sample at which conductance was measured

- The Foxboro Century OVA 128 will be calibrated to a methane in air standard (approximately 100 ppm) daily to ensure total volatile organic readings are accurate. If calibration problems are encountered, calibration will become more frequent as required. The methane in air standard is manufactured by Liquid Carbonic and is marked with its certified concentration. The standard is run directly into the intake of the pickup probe and the gain adjustment of the OVA 128 is then used to calibrate the reading to the standard concentration. Any OVA, total volatile organic readings will be reported as "X ppm as methane."

7.0 ANALYTICAL PROCEDURES

All analytical procedures to be used are officially approved EPA procedures. The appropriate methods and required holding times to be met are given in Table 4-1.

The primary compound list will be the Target Compound List (TCL) and the Target Analyte List (TAL). The analytical methods which are to be used for the analysis of the sample media collected at the Site will be in accordance with the most current "USEPA Contract Laboratory Program (CLP) Statement of Work (SOW) for Organic (and Inorganic) Analyses, OLM02 (and ILM02)".

Select samples will also be analyzed for PCB constituents: Total Suspended Solids (TSS), Total Dissolved Solids (TDS), Alkalinity (Total Carbonate, Phenolphthalein), BIO Chemical Oxygen Demand (BOD), Chemical Oxygen Demand (COD), Total Organic Carbon (TOC), sulfate, and ammonia-nitrogen using methods specified in "Test Methods for Evaluating Solid Waste", SW-846 third edition, November 1986, updated July 1992, and "Methods for Chemical Analysis of Water and Wastes", EPA-600/4-79-020, revised March 1983. These analytical methods were chosen to provide comparability with other data typically collected for CERCLA investigations and to meet the project DQOs. These methods are the most appropriate to achieve all DQOs. Attachment 1 presents details on the analytical methods.

Currently the only anticipated PCB analyses will be for the subsurface soil, surficial soil, waste, and sediment samples collected from Quarries No. 2 and No. 3. The remaining sample aliquots for PCB analyses will be extracted and the extracts stored pending the Quarries No. 2 and No. 3 analysis results. If PCB constituents are detected, then the remaining sample extracts will be analyzed.

Table 7-1 presents the list of organic, inorganic, and miscellaneous constituents and their respective quantitation limits (CRQLs/CRDLs/QLs) for the investigation. The TCL volatile and semivolatile organic fractions will also include mass spectral library searching for up to 10 additional volatile and 20 additional semivolatile, non-target (non-TCL) compounds.

Geotechnical testing will be performed using ASTM and SW-846 methods. Method references for the geotechnical testing are presented in Table 7-2.

**Table 7-1 Crater Resources PRP Site
Organic and Inorganic Constituents for Analysis and Quantitation /
Detection Limits (CRQL/CRDL/QL)**

Volatiles	CAS Number	Quantitation Limits ^a	
		Low Water µg/L	Low Soil/ Sediment ^b µg/Kg
1. Chloromethane	74-87-3	10	10
2. Bromomethane	74-83-9	10	10
3. Vinyl Chloride	75-01-4	10	10
4. Chloroethane	75-00-3	10	10
5. Methylene Chloride	75-09-2	10	10
6. Acetone	67-64-1	10	10
7. Carbon Disulfide	75-15-0	10	10
8. 1,1-Dichloroethene	75-35-4	10	10
9. 1,1-Dichloroethane	75-35-3	10	10
10. 1,2-Dichloroethene (total)	540-54-0	10	10
11. Chloroform	67-66-3	10	10
12. 1,2-Dichloroethane	107-06-2	10	10
13. 2-Butanone	78-93-3	10	10
14. 1,1,1,1,1-trichloroethane	71-55-6	10	10
15. Carbon Tetrachloride	56-23-5	10	10
16. Bromodichloromethane	75-27-4	10	10
17. 1,1,2,2-Tetrachloroethane	79-34-5	10	10
18. 1,2-Dichloropropane	78-87-5	10	10
19. cis-1,3-Dichloropropene	10061-01-5	10	10
20. Trichloroethene	79-01-6	10	10
21. Dibromochloromethane	124-48-1	10	10
22. 1,1,2-1,1,1-trichloroethane	79-00-5	10	10
23. Benzene	71-43-2	10	10
24. trans-1,3-Dichloropropene	10061-02-6	10	10
25. Bromoform	75-25-2	10	10
26. 2-Hexanone	591-78-6	10	10
27. 4-Methyl-2-Pentanone	108-10-1	10	10
28. Tetrachloroethane	127-18-4	10	10
29. Toluene	108-88-3	10	10

Table 7-1. Continued

Quantitation Limits ^a			
Volatiles	CAS Number	Low Water µg/L	Low Soil/ Sediment ^b µg/Kg
30. Chlorobenzene	108-90-7	10	10
31. Ethyl Benzene	100-41-4	10	10
32. Styrene	100-42-5	10	10
33. Total Xylenes	100-42-5	10	10

^a. Specific quantitation limits are highly matrix dependent. The quantitation limits which are listed may not always be achievable.

^b. Quantitation limits for soil/sediment are based on wet weight. Individual sample quantitation limits will be different based on dry weight correction. Medium Level Soil/Sediment Contract Required Quantitation Limits (CRQL) for Volatile TCL Compounds are 125 times the individual Low Level Soil/Sediment CRQL.

Table 7-1 Continued

Semivolatiles	CAS Number	Quantitation Limits ^a	
		Low Water µg/L	Low Soil/ Sediment ^c µg/Kg
34. Phenol	108-95-2	10	330
35. bis(2-Chloroethyl)ether	111-44-4	10	330
36. 2-Chlorophenol	95-57-8	10	330
37. 1,3-Dichlorobenzene	541-73-1	10	330
38. 1,4-Dichlorobenzene	95-50-1	10	330
39. 1,2-Dichlorobenzene	95-50-1	10	330
40. 2-Methylphenol	95-48-7	10	330
41. bis(2-Chloroisopropyl) ether	108-60-1	10	330
42. 4-Methylphenol	106-44-5	10	330
43. N-Nitroso-Di-n-propylamine	621-64-7	10	330
44. Hexachloroethane	67-72-1	10	330
45. Nitrobenzene	98-95-3	10	330
46. Isophorone	78-59-1	10	330
47. 2-Nitrophenol	88-75-5	10	330
48. 2,4-Dimethylphenol	105-67-9	10	330
49. bis(2-Chloroethoxy)methane	111-91-1	10	330
50. 2,4-Dichlorophenol	120-83-2	10	330
51. 1,2,4-Trichlorobenzene	120-82-1	10	330
52. Naphthalene	91-20-3	10	330
53. 4-Chloroaniline	106-47-8	10	330
54. Hexachlorobutadiene	87-68-3	10	330
55. 4-Chloro-3-methylphenol	59-50-7	10	330
56. 2-Methylnaphthalene	91-57-6	10	330
57. Hexachlorocyclopentadiene	77-47-4	10	330
58. 2,4,6-Trichlorophenol	88-06-2	10	330
59. 2,4,5-Trichlorophenol	95-95-4	25	800
60. 2-Chloronaphthalene	91-58-7	10	330
61. 2-Nitroaniline	88-74-4	25	800
62. Dimethyl Phthalate	131-11-3	10	330
63. Acenaphthylene	208-96-8	10	330
64. 3-Nitroaniline	99-09-2	25	800
65. Acenaphthene	83-32-9	10	330
66. 2,4-Dinitrophenol	51-28-5	25	800
67. 4-Nitrophenol	100-02-7	25	800
68. Dibenzofuran	132-64-9	10	330
69. 2,4-Dinitrotoluene	121-14-2	10	330
70. 2,6-Dinitrotoluene	606-20-2	10	330

Table 7-1 Continued

Semivolatiles	CAS Number	Quantitation Limits ^a	
		Low Water µg/L	Low Soil/ Sediment ^c µg/Kg
71. Diethylphthalate	84-66-2	10	330
72. 4-Chlorophenyl Phenyl ether	7005-72-3	10	330
73. Fluorene	86-73-7	10	330
74. 4-Nitroaniline	100-01-6	25	800
75. 4,6-Dinitro-2-methylphenol	534-52-1	25	800
76. N-nitrosodiphenylamine	86-30-6	10	330
77. 4-Bromophenyl Phenyl ether	101-55-3	10	330
78. Hexachlorobenzene	118-74-1	10	330
79. Pentachlorophenol	87-86-5	25	800
80. Phenanthrene	85-01-8	10	330
81. Carbazole	86-74-8	10	330
82. Anthracene	120-12-7	10	330
83. Di-n-butylphthalate	84-74-2	10	330
84. Fluoranthene	206-44-0	10	330
85. Pyrene	129-00-0	10	330
86. Butyl Benzyl Phthalate	85-68-7	10	330
87. 3,3'-Dichlorobenzidine	91-94-1	10	330
88. Benzo(a)anthracene	56-55-3	10	330
89. bis(2-ethylhexyl)phthalate	117-81-7	10	330
90. Chrysene	218-01-9	10	330
91. Di-n-octyl Phthalate	117-84-0	10	330
92. Benzo(b)fluoranthene	205-99-2	10	330
93. Benzo(k)fluoranthene	207-08-9	10	330
94. Benzo(a)pyrene	50-32-8	10	330
95. Indeno(1,2,3-cd)pyrene	193-39-5	10	330
96. Dibenz (a,h)anthracene	53-70-3	10	330
97. Benzo(g,h,i)perylene	191-24-2	10	330

^c Quantitation limits for soil/sediment are based on wet weight. Individual sample quantitation limits will be different based on dry weight correction. Medium Soil/Sediment Contract Required Quantitation Limits (CRQL) for Semivolatile TCL Compounds are 30 times the individual Low Soil/Sediment CRQL.

Table 7-1 Continued

Pesticides/PCBs	CAS Number	Quantitation Limits ^a	
		Low Water µg/L	Low Soil/ Sediment ^d µg/Kg
98. Aroclor-1016	12674-11-2	1.0 ✓	33.0
99. Aroclor-1221	11104-28-2	2.0 ✓	67.0
100. Aroclor-1232	11141-16-5	1.0 ✓	33.0
101. Aroclor-1242	53469-21-9	1.0 ✓	33.0
102. Aroclor-1248	12672-29-6	1.0 ✓	33.0
103. Aroclor-1254	11097-69-1	1.0 ✓	33.0
104. Aroclor-1260	11086-82-5	1.0 ✓	33.0

^d Quantitation limits for soil/sediment are based on wet weight. Individual sample quantitation limits will be different based on dry weight correction. Medium Level Soil/Sediment Contract Required Quantitation Limits (CRQL) for Pesticide/PCB TCL Compounds are 30 times the individual Low Level Soil/Sediment CRQL.

Table 7-1 Continued

**Elements Determined by Inductively Coupled
Plasma Emission or Atomic Absorption Spectroscopy**

Element (TAL Inorganics)	Contract Required Detection Limit Water (µg/L)	Contract Required Detection Limit Soil ^e (mg/Kg)
Aluminum	200	40
Antimony	60	12
Arsenic	10	2
Barium	200	40
Beryllium	5	1
Cadmium	5	1
Calcium	5000	1000
Chromium	10	2
Cobalt	50	10
Copper	25	5
Iron	100	20
Lead	3	0.6
Magnesium	5000	1000
Manganese	15	3
Mercury	0.2	0.1
Nickel	40	8
Potassium	5000	1000
Selenium	5	1
Sodium	5000	1000
Silver	10	2
Thallium	10	2
Vanadium	50	10
Zinc	20	4
Cyanide	10	0.5

^e Soil CRDL's presented are based on wet weight. Individual sample detection limits will be different based on dry weight correction.

Table 7-1 Continued

Miscellaneous Parameters

Analyte	Quantitation Limit	Quantitation Limit
	Water (mg/L)	Solid (mg/Kg)
Ammonia-nitrogen	0.04	5
Sulfate	10.0	25
Total Suspended Solids (TSS)	1.0	NA
Total Dissolved Solids (TDS)	10.0	NA
Alkalinity (Total Carbonate, Phenolphthalein)	1.0	NA
Biochemical Oxygen Demand (BOD)	2.0	NA
Chemical Oxygen Demand (COD)	10.0	10
Hardness	1.0	NA
Total Organic Carbon (TOC)	0.5	5

* Soil QL's presented are based on wet weight. Individual sample quantitation limits will be different based on dry weight correction.

NA - Not Analyzed.

Table 7-2 *Method Reference Numbers for the Geotechnical Testing*

Test	Method Number
Grain Size Distribution	ASTM D422
Hydrometer	
Moisture Contents	ASTM D2216

8.0

DATA REPORTING, VALIDATION, AND REDUCTION

Data validation practices will be followed to ensure that raw data are not altered and that an audit trail is developed for those data which required reduction. All the field data, such as those generated during field measurements, observations, and field instrument calibrations, will be entered directly into a bound field notebook. Each project team member will be responsible for proofing all data transfers made, and the RI Site Manager will proof at least ten percent of all data transfers.

Upon receipt of the sample data packages, the laboratory data will be quantitatively and qualitatively validated by ERM's Quality Assurance Chemist. Data validation is discussed in detail in Section 12.0.

It is anticipated that ERM's data reduction for this investigation will be minimal and will consist primarily of tabulating IEA's analytical results onto summary tables through the use of computerized spreadsheet software. All IEA analytical data will be provided in the form of diskette deliverables. This data will be loaded into ERM's GIS database. All reduced data will be assigned document control identification numbers and placed in the central file maintained by the Project Manager.

Analytical data for soil/solid matrices will be reported as $\mu\text{g}/\text{Kg}$ (TCL parameters) and mg/Kg (TAL and miscellaneous parameters). Analytical data for aqueous matrices will be reported as $\mu\text{g}/\text{L}$ (TCL/TAL parameters) and mg/L (miscellaneous parameters) with the exception of total organic carbon. Analysis results for this parameter will be expressed in terms of "percent (%) organic carbon". Data packages associated with the TCL/TAL analyses of samples collected during the investigation will be prepared utilizing US EPA CLP deliverable formats. Analytical data for miscellaneous parameters will be reported as mg/L . The deliverable requirements for the non-CLP parameters are presented in Table 8-1. Results of the geotechnical testing will be reported as specified in the method referenced.

All raw field data will be summarized, reduced, or tabulated for use in the investigation reports by the RI Site Manager. All laboratory analytical data will be summarized and tabulated upon receipt, validated and qualified (see Section 12.0), and the final data submitted to the project team for use in the investigation reports.

ERM will require a rigorous data control program which will ensure that all documents for the investigations are accounted for as they are completed. Accountable documents include items such as logbooks, field data records, correspondence, chain-of-custody records, analytical reports, data packages, photographs, computer disks, and reports. The Project Manager is responsible for maintaining a central file in which all accountable documents will be inventoried.

To maintain control in the transfer of data, all copies of raw data from the field notebooks, and the data as received from the laboratory, will be entered into a data file and assigned an appropriate document control identification number. The data file will serve as the ultimate archive for all information and data generated during this investigation.

The documentation of sample collection will include the use of bound field logbooks in which all information on sample collection and field instrument calibration will be entered in indelible ink. Appropriate information will be entered to reconstruct the sampling event, including site name (top of each page), sample identification, brief description of sample, date and time of collection, sampling methodology, field measurements and observations, and sampler's initials (bottom of each page with date). ERM's Sampling Notebook SOP is photocopied and attached to the front cover of all field books issued. This is presented as Figure 8-1.

Table 8-1 Required Deliverables for CLP-Equivalent Format

- **Title Page** - present site name, field sample numbers and corresponding laboratory control numbers and the appropriate laboratory manager's signature authorizing release of the data.
- **Case Narrative** - summarize any problems encountered during analysis and discuss any corrective actions taken.
- **Table of Contents** - list all major sections of the delivered document with the referenced page numbers. This can be incorporated onto the Title Page.
- **ERM Chain of Custody Forms and Traffic Report Forms** - copies of the documents signed by the laboratory sample log-in personnel.
- **Laboratory Chronicle** - supply the dates of preparation and analysis for each analysis fraction and sample.
- **Methodology Summary** - present a brief summary of the method used and the appropriate method reference.
- **Analysis Reports** - present the analyte and indicate the values for positive hits, the quantitation limit, and moisture content for soil samples. Report an individual analysis report for each sample. Soil sample results must be reported on a dry weight basis.
- **Quality Control Summaries** - for GC analyses, present summary forms of surrogate compound recoveries, matrix spike/matrix spike duplicate recoveries, quality control sample analysis results, calibration standard data, retention time data, and method blank analysis results.

For miscellaneous parameter analyses, present summary forms of laboratory duplicate and matrix spike sample analysis results, laboratory control sample analysis results, and laboratory method blank results.

Table 8-1 Required Deliverables for CLP-Equivalent Format (continued)

GC Analysis Data

- **Raw Sample Data** - provide GC chromatograms and instrument quantitation reports which include library list of compounds, peak retention times, peak areas, peak heights and raw concentration data. Chromatograms must be provided for both primary and confirmation columns. Provide soil sample extraction or preparation logs. Provide an example of how positive results are calculated.
- **Standards Data Package**
 - **Initial Calibration** - provide GC chromatograms and instrument quantitation reports which include library list of compounds, peak retention times, peak areas, peak heights and raw concentration data for each level standard associated with the initial calibration. Provide response factors for each standard.
 - **Continuing Calibration** - provide GC chromatograms and instrument quantitation reports which include library list of compounds, peak retention times, peak areas, peak heights and raw concentration data for all check standards. Provide response factors and indicate if acceptance criteria was met for the continuing calibration.
 - **Raw Quality Control Data** - provide GC chromatograms and instrument quantitation reports which include library list of compounds, peak retention times, peak areas, peak heights and raw concentration data for each method blank and the matrix spike and matrix spike duplicate.
 - **Miscellaneous Quality Control Data** - Run logs presenting the chronology of sample and standard analysis and surrogate retention time shifts must be provided for both the primary and confirmation columns.

Miscellaneous Parameter Data

- **Raw Sample Data** - provide instrument printouts or photocopies of laboratory notebook records of raw sample data. Provide soil sample extraction or preparation logs.
- **Standards Data** - provide instrument printout or laboratory notebook records associated with the initial and continuing calibrations.

Figure 8-1 Sampling Logbook SOP

One or more bound books will be maintained for each site; the book(s) will remain with the site evidence file. Copies should be made for the person who made the entries and the PM if requested.

All entries in the Logbook must be made in ink.

First Page should contain:

- Site name and number
- Date and time started
- Personnel on-site

Next page(s) DTW for all wells if required by the sampling plan. S/N of the DTW meter.

Each new day should contain:

- Date and time started
- Weather
- Personnel on-site — including any non-ERM personnel
- Sampling information (see next page)

- * When a mistake is made in the Log, put a single line through it in ink and initial and date.

Figure 8-1 Sample Information

- Sample # (Traffic Report)
- Date and Time Sample collected
- Source of Sample (well, stream, domestic well, field etc.)
- Purged Well - type of equipment, purge volume, rate of purge, and decon procedures.
- Location of Sample - document with a site sketch and/or written description, where sample was taken so that it could be found again.
- How was sample taken? (bailer, trowel, SS spoon, thief, etc.)
- Analysis and QA/QC required (601, 602, Metals, Tier I, Tier II, etc.)
- Chemical Preservation used (HNO₃, H₂SO₄, NaOH, etc.)
- Field instrument calibration including date of calibration, standards used and their source, results of calibration and any corrective actions taken.
- Field Data (pH, DO, spec. conductance, temp., etc.)
- Field Observations - significant observation should be documented.
 - Sample condition (color, odor, turbidity, oil, sheen)
 - Site condition (stressed vegetation, exposure of buried wastes, erosion problems, etc.)
- How sample was shipped, date, time and where to, and if legal seals were attached to transport container(s).
- Comments - Any observation or event that occurred that would be relevant to the site; for example, weather changes or effect it had on sampling, conversations with the client, public official or private citizen; instrument calibration, equipment problems, etc.

9.0 **INTERNAL QUALITY CONTROL CHECKS**

Internal quality control checks for the analytical laboratory and the field are presented below. The data quality objective criteria for these internal quality control checks are presented in Table 3-2.

9.1 **LABORATORY INTERNAL QUALITY CONTROL CHECKS**

IEA's Internal Quality Control Checks are presented in Attachment 1. These will be a continuation of ERM's Field Internal Quality Control Checks presented below.

9.2 **FIELD INTERNAL QUALITY CONTROL CHECKS**

Field Internal Quality Control Checks will be utilized during this investigation through the use of the following:

- **Trip Blanks** - These blanks consist of ultrapure, deionized water contained in each sample container. These blanks will accompany the samplers during the sampling process and will serve as QC checks on container cleanliness, external contamination, and the analytical method. Trip blanks will be submitted blind using a fictitious sample location one per cooler for volatiles analysis for subsurface soil, surficial soil, surface water, sediment, and ground water samples.
- **Equipment Rinsate Blank** - Equipment rinsate blanks will be collected to ensure that sampling equipment is clean and that the potential for cross contamination has been minimized by the equipment decontamination procedures. These blanks will be collected by decontaminating the sampling device and then pouring ultrapure deionized water over the device. This rinsate water will be collected into a clean stainless steel bowl and then transferred to the appropriate sample containers. One equipment rinsate blank will be collected for each sampling device associated with the subsurface soil, surficial soil, surface water, sediment, and ground water samples. Solid sampling equipment rinsate blanks will be collected from split spoon samplers, hand augers, spoons, etc. The equipment rinsate blanks will be analyzed for identical parameters as the samples. Because dedicated bailers will be utilized for ground water sampling, the equipment blanks will be collected for dissolved metals analyses

only, and will consist of ultrapure deionized water through the Millipore® filtration apparatus.

- **Ambient Blank** - Ambient blanks will be collected to ensure that sample containers are clean. These blanks will be collected by pouring ultrapure deionized water into the sample container in the field. Ambient blanks will be collected for each media sampling event at a frequency of one per twenty samples or one per sampling event, whichever is more frequent. The ambient blanks will be analyzed for identical parameters as the samples.
- **Duplicate Samples** - Blind duplicate samples will be collected to allow determination of analytical and sampling precision. One duplicate sample in every twenty subsurface soil, surficial soil, surface water, sediment, and ground water samples will be collected and submitted for the identical parameters as the true sample.
- **Matrix Spike Sample** - Matrix spike/matrix spike duplicate (MS/MSD) samples will also be submitted as further QC checks. These samples will be spiked by the laboratory. These will be collected at the frequency of one MS and MSD for every twenty field samples (including trip blanks, field blanks, and blind duplicates). These will allow accuracy to be determined by the recovery rates of compounds (the matrix spike and/or surrogate spike compounds defined in the analytical methods). Precision will also be assessed by comparison of matrix spike and matrix spike duplicate recoveries. The purpose of these laboratory spikes is to monitor any possible matrix effects specific to samples collected from the Crater Resources site. The addition of known concentrations of compounds/constituents into the sample also monitors extraction/digestion efficiency.

Matrix spike/matrix spike duplicate sample aliquots for solid/soil analyses will be split from the designated sample at the laboratory. The laboratory will select aliquots that are as homogeneous with respect to one another as possible to avoid precision problems related to sample inhomogeneity. The specific sample locations which will be sampled for matrix spike and blind duplicate analyses will be chosen by the RI Site Manager with direction from the Quality Assurance Manager. Homogenizing is discouraged prior to splitting duplicates to avoid the loss of volatile organic compounds.

10.0 PERFORMANCE AND SYSTEMS AUDITS

10.1 ON-SITE AUDIT

One on-site systems field audit will be performed during the subsurface soil sampling event to review all field-related quality assurance activities. The system audits will be conducted by ERM's Quality Assurance Manager or the Field Services Manager. Figure 10-1 presents ERM's Quality Assurance Audit forms. The acceptance criteria for the field audit will be in adherence to the protocols presented throughout the QAPP. Deficiencies found during the audits will be brought to the attention of the responsible individuals and corrective action as per Section 13.0 of this document will be initiated. Copies of the audits will be distributed to all project personnel key,

Specific elements of the on-site audit include the verification of the following:

- Completeness and accuracy of sample Chain-of-Custody forms, including documentation of times, dates, transaction descriptions, and signatures.
- Completeness and accuracy of sample identification labels, including notation of time, date, location, type of sample, person collecting sample, preservation method used, and type of testing required.
- Completeness and accuracy of field notebooks, including documentation of times, dates, drillers' names, sampling methods used, sampling locations, number of samples collected, name of person collecting samples, types of samples, results of field measurements, soil logs, and any problems encountered during sampling.
- Adherence to health and safety guidelines outlined in the Site Health and Safety Plan including wearing of proper protective clothing.
- Adherence to decontamination procedures outlined in Section 4.0 of this QAPP, including proper documentation of pumps and pump tubing, bailers, and sampling equipment.
- Adherence to sample collection, preparation, preservation, and storage procedures.

10.2 LABORATORY AUDIT

10.2.1 *Internal Laboratory Audits*

IEA performs regular systems and performance audits, and these are described in Attachment 1.

10.2.2 *ERM's Performance Audit of IEA*

IEA is not a participant in EPA's Contract Laboratory Program (CLP). An on-site audit will be performed by ERM while soil samples collected as part of the RI are in-house to ensure adherence to the QAPP.

Results of the evaluation of both the field and laboratory audits will be submitted to ERM's Project Manager for review and incorporation into the following months' reports.

If the results of the audit necessitate further action, the Project Manager will be notified of such and will be apprised of any corrective action taken.

10.3 GEOTECHNICAL LABORATORY PERFORMANCE AND SYSTEM AUDITS

Geotechnical laboratory performance and system audits are described in Attachment 2.

Figure 10-1 ERM Quality Assurance Audit Form

Project: _____ WO Number: _____

Date: _____

Auditor(s): _____

On-Site Sampling Personnel: _____

Audit Conducted on the following:

☐ Soil Sampling

☐ Surface Water/Sediment

☐ Ground Water

☐ Decontamination

Y = Yes

N = No

N/A = Not Applicable

N/D = Not Determined

Sample Collection:

Do sampling locations agree with those specified in the Work Plan/Sampling Plan? _____

Is the sampling location either documented sufficiently and marked to allow it to be found/sampled again in the future? _____

Are sampling times, ERM Traffic Report Numbers and sample description noted in the FNB? _____

Is sampling proceeding from the suspected least contaminated area to the most contaminated area? _____

Have all field measurements been properly taken as per Sampling Plan? _____

Are field measurement(s) being taken immediately after the sample is collected? _____

Have sample bottles been labeled properly? _____

Have proper containers and preservatives been used? _____

Are proper sample volumes procured? _____

Does the potential for sample cross-contamination exist based on procedures observed? _____

Have MS and MSD(s) been collected as per QA/QC Plan? _____

Does a travel blank exist for each matrix present? _____

Are samples being refrigerated/iced immediately after collection? _____

Has condition of sample been recorded in the FNB and in the traffic report? _____

Have legal seal(s) been properly filled out and attached to the shipping container(s)? _____

Figure 10-1 Continued

Soil Sampling (Check if not applicable):

Type: _____ Hand: _____ Auger or Rig-Backhoe Pit

Are samples being collected at proper depths?

Are samples being screened with an OVA (if specified in Work Plan and applicable)?

Is a description of soils/materials being logged?

Have soils been homogenized where applicable (Specified by the Sampling Plan)?

Surface Water/Sediment Sampling (check if not applicable):

Have stream flow and velocity parameters been noted?

☐ Estimated or ☐ Measured

Has sampling proceeded from downstream to upstream locations?

Has the sampler acquired the water sample upstream of his position to minimize suspended sediment from entering the sample?

Have water samples been collected in the mixing zone, not stagnant areas?

Have sediments been characterized as to type and size distribution?

Has the proper sediment fraction (fine, depth) been sampled for the analyses of interest?

Ground Water Sampling (Check if not applicable):

Have organic vapor readings been obtained when the well head was opened?

Have depth to water level readings been taken for all wells?

Have the well specifications been noted properly (i.e., total depth, casing diameter, depth-to-water to the nearest one-hundredth of a foot, etc.)?

Has the purge volume been calculated properly?

Has well yield been properly evaluated to determine when sample acquisition should take place (i.e., does well go dry and need to recover)?

Has the purge pump been placed at the proper level to ensure proper well evacuation?

Figure 10-1 Continued

What evacuation method has been used? _____

- ☐ Bailer ☐ Submersible ☐ Other _____
☐ Bladder Pump ☐ Centrifugal Pump ☐ Fultz Pump

If metals are being analyzed, have the samples been field filtered? _____

Are field pH, conductivity, and temperature being measured and documented? _____

Is there documentation of calibrating the instruments? _____

Are bailer bags marked as to site name, well ID, and date of dedication? _____

Is bailer line and bailer dedicated to each well and line disposed of after use? _____

Bailer Type _____ Line Type _____

Have appropriate measures been taken to dispose of contaminated purge water, pump lines, bailers, etc.? _____

For Domestic Wells - Has as much information on the well and distribution system been obtained (i.e., depth, casing type, diameter, treatment present, etc.)? _____

Has the sample been collected prior to treatment and as close to the well head as possible? _____

Has the domestic well been purged sufficiently to reach pH, conductivity, and temperature stabilization? _____

Have any fixtures been removed from the domestic well before the sample was taken? _____

Decontamination:

Has sampling equipment been decontaminated properly for the given analytes as per QA Plan? _____

Have the proper decontamination solutions been used? _____

For large equipment (backhoes, drill rigs), has decontamination taken place in an appropriate area? _____

Has decontamination water/solution been collected for proper disposal? _____

Where disposed? _____

Figure 10-1 Continued

Has disposable equipment, that is contaminated, been properly decontaminated and disposed of? _____

Have decon samples been taken from the sampling equipment as per Sampling Plan? _____

Has all appropriate information been recorded in the FNB? _____

Have the weather conditions been recorded? _____

Are weather conditions affecting sample quality? _____

Is the "Chain of Custody" being maintained for the samples? _____

Have all personnel been properly trained to operate the equipment present? _____

Are the objectives of the sampling activities understood by the field personnel? _____

Are employees conducting the investigation in a professional manner? _____

Audit Summary and Comments:

Signed by:

Sampler: _____ Print Name: _____

Auditor: _____

Date: _____

11.0 PREVENTIVE MAINTENANCE

11.1 LABORATORY MAINTENANCE

IEA's laboratory preventive maintenance programs and requirements are described in Attachment 1.

11.2 FIELD MAINTENANCE

ERM's field equipment is maintained through the use of a tracking system incorporating the tagging of each equipment item. This tag identifies its most recent maintenance, battery charge, and condition. When damaged or equipment in need of repair is returned to the equipment warehouse, it is appropriately flagged for the required maintenance to be performed. This process ensures that only operable and maintained equipment enters the field. Routine daily maintenance procedures conducted in the field will include the following:

- Removal of surface dirt and debris from exposed surfaces of the sampling equipment and measurement systems.
- Cleansing of filters on the organic vapor analyzer.
- Storage of equipment away from the elements.
- Daily inspections of sampling equipment and measurement systems for possible problems (e.g., cracked or clogged lines or tubing or weak batteries).
- Checking of instrument calibrations as described in Section 6.2 of this QAPP.
- Charging any battery packs for equipment when not in use.

Spare and replacement parts stored in the field to minimize downtime include the following:

- Appropriately sized batteries
- Locks
- Extra sample containers and preservatives
- OVA igniters and filters

- OVA H2 gas, battery charger, and support equipment
- Extra samples coolers, packing material, and sample location stakes.
- Additional supply of health and safety equipment, i.e., respirator cartridges, boots, gloves, tyvek, etc.
- Additional equipment as necessary for the field tasks.

11.3 **GEOTECHNICAL LABORATORY PREVENTIVE MAINTENANCE**

The geotechnical laboratory preventative maintenance requirements are described in Attachment 2.

12.0 SPECIFIC ROUTINE PROCEDURES USED TO ASSESS DATA PRECISION, ACCURACY, AND COMPLETENESS

12.1 OVERALL PROJECT ASSESSMENT

Overall data quality will be assessed by a thorough understanding of the data quality objectives which are stated during the design phase of the investigation. By maintaining thorough documentation of all decisions made during each phase of sampling, performing field and laboratory audits, thoroughly reviewing (validating) the analytical data as it is generated by the laboratory, and providing appropriate feedback as problems arise in the field or at the laboratory, ERM will closely monitor data accuracy, precision, and completeness.

12.2 FIELD QUALITY ASSESSMENT

To ensure that all field data are collected accurately and correctly, specific written instructions will be issued to all personnel involved in field data acquisition by the Project Manager. The Field Services Coordinator will perform field audits during the initial sampling events of the investigation to document that the appropriate procedures are being followed for sample (and blank) collection. These audits will include a thorough review of the field books used by the Project Personnel to ensure that all tasks were performed as specified in the instructions. The field audits will necessarily enable the data quality to be assessed with regard to the field operations.

The evaluation (data review) of trip and field blanks and other field QC samples will provide definitive indications of the data quality. If a problem arises which can be isolated, corrective actions can be instituted for future field efforts.

12.3 LABORATORY DATA QUALITY ASSESSMENT

Specific measures that will be taken by IEA to assess data quality are presented in Attachment 1.

12.4 ERM'S LABORATORY DATA ASSESSMENT

12.4.1 ERM Data Validation

All analytical data generated during the investigation will undergo a rigorous ERM data review. This review will be performed in accordance with the most current Region III modifications to "Laboratory Data Validation Functional Guidelines for the Evaluation of Organic (and Inorganic) Analysis" (US EPA Data Review Work Group, 6/91 for Organics and 7/88 for Inorganics).

A preliminary review will be performed to verify all necessary paperwork (chain-of-custodies, traffic reports, analytical reports, laboratory personnel signatures) and deliverables as stated in the CLP requirements are present.

A detailed quality assurance review will be performed by the ERM Quality Assurance Chemist to verify the qualitative and quantitative reliability of the data as it is presented. This review will include a detailed review and interpretation of all data generated by IEA. The primary tools which will be used by experienced data review chemists will be guidance documents, established (contractual) criteria, and professional judgment. Table 12-1 presents the items examined during the quality assurance review.

Based upon the review of the analytical data, an organic and inorganic quality assurance report will be prepared which will state in a technical, yet "user friendly" fashion the qualitative and quantitative reliability of the analytical data. The report will consist of a general introduction section, followed by qualifying statements that should be taken into consideration for the analytical results to best be utilized. Based upon the quality assurance review, qualifier codes will be placed next to specific sample results on the sample data tables. These qualifier codes will serve as an indication of the qualitative and quantitative reliability of the data.

During the course of the data review, an organic and inorganic support documentation package is prepared which will provide the backup information that will accompany all qualifying statements presented in the quality assurance review.

Once the review has been completed, the Quality Assurance Manager will verify the accuracy of the review and will then submit these data to the Project Manager. These approved data tables and quality assurance reviews will be signed and dated by the Quality Assurance Manager.

12.5 DATA MANAGEMENT QUALITY ASSESSMENT

As the analytical data generated from the subject investigation are validated, qualified, and submitted to the Project Manager, the quality of the data will be assessed from an overall management perspective by direct comparison of analytical results obtained from previous samplings. Information that can be obtained includes comparison of results obtained from samples taken within the same general vicinity, and the identification of missing data points. By examination of the data at the "back-end" of the process, the data quality can be assessed with respect to representativeness, precision, compatibility, and completeness.

Table 12-1 Items Reviewed During the ERM Data Validation

Area Examined	Applicability (organic, inorganic, both)
ERM and Laboratory Chain of Custodies (Traffic Reports, Field Notes, etc.)	Both
Holding Times	Both
Extraction/Digestion Logs	Both
Blanks - field and laboratory (accuracy)	Both
Instrument Tune	Organic
Standards	Both
Linearity	Both
Sensitivity/Stability	Both
Selectivity/Specificity	Both
EPA Criteria (SPCC & LCS)	Both
Variability of Technique (internal standards)	Organic
Analytical Run Log	Inorganic
ICP Interference	Inorganic
Control Standards	Both
Samples	
Detection Limits	Both
Instrument Printouts	Both
ICP data	Inorganic
AA data	Inorganic
GC data	Organic
GG/MS data	Organic
Autoanalyzer data	Inorganic
Qualitative Identification	Both
Mass spectra	
PCBs results	
Tentatively identified compounds	
Quantitative Reliability	Both
Calculations/Equations	Both
Matrix spikes (accuracy)	Both
Bias	
Matrix spike duplicates	Organic
Bias	
Accuracy & Precision	
Surrogate Spikes	Organic
Bias	
Duplicates (field and laboratory)	Both
Precision	
Representativeness	
Post-Digestion Spikes	Inorganic
Matrix Effects	

13.0 CORRECTIVE ACTION

13.1 THE ANALYTICAL LABORATORY'S CORRECTIVE ACTION

Correction actions for IEA are presented in Attachment 1. IEA will provide documentation as to what, if any, corrective actions were initiated concerning this study and report them to ERM's Quality Assurance Manager.

13.2 ERM'S CORRECTIVE ACTION

Field quality assurance activities will be reported topically to ERM's Project Manager. Problems affecting quality assurance that are encountered during the study will be reported on a Corrective action Form as presented in Figure 13-1. The Project Manager will be responsible for initiating the corrective actions and for ensuring that the actions are taken in a timely manner and that the desired results are produced. The Project Manager will report to the Quality Assurance Manager and project team on all necessary corrective actions taken, the outcome of these actions, and their effect on data produced. All corrective action taken will be reported to Crater Resources.

13.3 GEOTECHNICAL LABORATORY CORRECTIVE ACTION

Results of quality assurance checks will be reported to the licensed professional engineer responsible for overseeing the activities of the geotechnical technicians. Problems affecting quality assurance will be documented on a Corrective Action Form as described above, and forward to the Project Manager. The Quality Assurance Manager will be informed of all necessary corrective actions taken, the outcome of these actions, and their effect on data produced.

Figure 13-1 Corrective Action Form

Date: _____

Job Name: _____

Initiator's Name and Title: _____

Problem Description: _____

Source of Defect: _____

Reported To: _____

Corrective Action: _____

Reviewed and Implemented By: _____

cc: Project Manager: _____

QA Manager: _____

14.0

QUALITY ASSURANCE REPORT TO MANAGEMENT

After project initiation the Project Manager, in conjunction with the Quality Assurance Manager, will submit a progress report summary of all applicable quality assurance activities. These summaries shall contain at least the following types of information:

- The status and coverage of various laboratory and field quality assurance project activities.
- Data quality assurance reviews including assessment of accuracy, precision, completeness, representativeness, and comparability.
- Significant quality assurance problems discovered, corrective actions taken, progress and improvements, plans, and recommendations for further implementation of updating of the investigative QAPP.
- Any significant field observations noted in the field notebook during the sampling procedure.
- Results of performance and system audit reports.

***Attachment 1
IEA Comprehensive Quality
Assurance Plan and Standard
Operating Procedures***

AR300071

**IEA Corporation
Whippany, New Jersey**

QUALITY ASSURANCE PROGRAM PLAN

prepared by

**Kevin J. Gorman
Quality Assurance Manager**

and

**James A. Ploscyca
Corporate Quality Assurance Director**

This document has been prepared by IEA Corporation and will be updated annually. The material contained herein is not to be disclosed to any third party without prior written approval by IEA - New Jersey

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QUALITY ASSURANCE PROGRAM PLAN**TABLE OF CONTENTS**

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1.0 Quality Assurance Program Plan Identification Form

Document Title: IEA Corporation Whippany, New Jersey Quality Assurance Program Plan

Corporate Address: IEA Corporation
3000 Weston Parkway
Cary, North Carolina 27513
(919) 677-0090

Company Official: Dr. Kyle E. Dolbow
Title: Director of Operations
Telephone: (201) 428-8181

Company Official: Mr. Kevin J. Gorman
Title: Quality Assurance Manager
Telephone: (201) 428-8181

Company Official: Dr. Deborah Smith
Title: Laboratory Director
Telephone: (201) 428-8181

Company Official: Mr. Brian W. Wood
Title: Organic Manager
Telephone: (201) 428-8181

Company Official: Mr. Patrick Brown
Title: Senior Project Manager
Telephone: (201) 428-8181

Company Official: Mr. Michael Stone
Title: Field Services Manager
Telephone: (201) 428-8181
Plan Coverage: IEA Corporation Laboratory in Whippany, New Jersey

Location: 628 Route 10
Whippany, New Jersey 07981

ctions: Environmental Testing
Quality Assurance

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Date: 11/01/93

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Concurrences:

Name: Dr. Kyle E. Dolbow
Title: Director of NJ Operations
Location: Whippany, New Jersey

Signature: Kyle E. DolbowDate: 11/30/93

Name: Mr. Kevin J. Gorman
Title: Quality Assurance Manager
Location: Whippany, New Jersey

Signature: Kevin J. GormanDate: 11/30/93

Name: Dr. Deborah Smith
Title: Laboratory Director
Location: Whippany, New Jersey

Signature: Deborah SmithDate: 11/30/93

Name: Mr. Brian W. Wood
Title: Organic Manager
Location: Whippany, New Jersey

Signature: Brian W. WoodDate: 12/1/93

Name: Mr. Patrick Brown
Title: Senior Project Manager
Location: Whippany, New Jersey

Signature: Patrick M. BrownDate: 11/30/93

Name: Mr. Michael Stone
Title: Field Services Manager
Location: Whippany, New Jersey

Signature: Michael StoneDate: 12/2/93

Name: Mr. James A. Ploscyca
Title: Corporate Quality Assurance Director
Location: Cary, North Carolina

Signature: James A. PloscycaDate: 12/8/93

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2.0 Introduction

IEA, Inc. of New Jersey believes that it has the responsibility of producing quality analytical data, on time, which conforms to government regulations and is legally valid. It is our belief that through careful quality-conscious planning, we can maintain a sound client relationship, execute high quality work in a timely manner, and help safeguard the environment for the future.

It is realized that quality can only be achieved through its planning, implementation and final assessment stages of a project. Furthermore, management recognizes that it must communicate the quality of work expected from each employee. The quality assurance program described below has been established and applies to all scientific and technical work.

The purpose of this document is to present, in generalized terms, the policies, organizational structure, objectives, functional activities, and QA/QC procedures performed at IEA - NJ.

3.0 Quality Assurance Policy Statement

It is the intention of IEA - New Jersey to consistently produce analytical data which are accurate, reliable and fully meet clients' data quality objectives. The contents of this Quality Assurance Program Plan describe the activities which are utilized in order to ensure this commitment is maintained.

IEA - New Jersey Quality Policy

"Management and staff are committed to maintaining a carefully controlled analytical environments in order to ensure the consistent generation of accurate data which meets or exceeds the data quality objectives of our clientele".

Detailed written analytical procedures are used to ensure strict adherence to published analytical methods throughout the laboratory. Bench-level quality control measures with well defined acceptance criteria are included in each analytical procedure employed by the laboratory. Laboratory records and quality control data are monitored by management on a regular basis.

This manual is a written description of the structures employed by IEA to ensure that all data generated by the laboratory is accurate and defensible.

4.0 Quality Assurance Management

4.1 Introduction

IEA Corporation's Quality Assurance department consists of a Quality Assurance Manager at each of the six network laboratories. The laboratory Quality Assurance Manager reports directly to the Director of Operations for that facility and indirectly to the Corporate Director of Quality Assurance.

4.2 Communications

The Quality Assurance department communicates internally and externally through various means. Communication can take place via telephone, memoranda, or take the form of audit reports. At the present time, the Quality Assurance department participates in a weekly conference call to discuss relevant issues and disseminate information.

4.3 Document Control

IEA maintains the central computer file at corporate headquarters which contain all files utilized by the corporation. The following conventions are used to organize these files.

All documents are assigned a unique document control number which is printed in the upper right hand corner of the document. The name will correspond to the standard PC file naming convention which allows for eight (8) characters and a three (3) digit extension. All document names will have a total of eleven (11) characters generating a unique identification for each document. IEA's document control procedure is detailed in Corporate Document #QAS00100.NET. A copy of this document is available for review during a site visit.

4.4 QA Program Assessment

Each Quality Assurance Manager prepares a monthly QA report for Corporate QA. Semi-annual system audits are performed by each Quality Assurance Manager according to a Corporate audit checklist. A copy of the audit report is distributed to both the Corporate QA Director and the facility Director of Operations. A summary of these reports are prepared each month by the Corporate QA Director and are submitted to upper management. A typical status report would include such information as:

- changes in the quality assurance program
- summary of proficiency results at each network laboratory
- changes in certification status
- summary of system audits conducted at each network laboratory
- accomplishments since the previous report
- summary of training

The Quality Assurance program can only accomplish its objectives if management and staff are committed to adherence to the program. In order to assess continued compliance and to identify strong and weak points of the program, the Network QA Director conducts semi-annual assessments at each location.

The primary objective of IEA is to provide legally and scientifically defensible data to clients in a timely and cost effective manner. The achievement of this goal is dependent upon an active Quality Assurance program. The Quality Assurance Manager is responsible for monitoring laboratory compliance with all elements of the Quality Assurance program and communicating the status of the program to the Corporate QA Director as well as the Director of Operations.

The Quality Assurance Officer is responsible for preparing regular reports to management which reflect the overall precision and accuracy of the lab. This report is addressed to the Director of Operations of IEA - NJ Inc. and the Corporate Quality

Assurance Director. A copy of this report is distributed to all facility Managers and Group Leaders. These reports are generated once a month, and include the following:

- Turnaround time reports
- External audit results
- Internal audit results
- Conformance to quality control guidelines and limits
- Corrective action reports
- Defect log reports
- Regulatory compliance issues
- Recommended or anticipated changes to the QA program
- Miscellaneous QA issues
- Summary of overall QA program

5.0 Personnel Qualifications

Personnel Qualifications

The laboratory is organized by analytical groups, with first-line responsibility at the Group Leader level and so on up the organizational ladder. This structure also represents the chain of command within the organization.

Corporate Management

Corporate Management is responsible for communicating their goals and requirements to the laboratory, they must set the tone for the level of quality that is expected in all day-to-day operations. Accurate communication of the mission statement, operating philosophy, ethics policy and quality assurance standards must occur as an on-going process. They must ensure that all personnel with direct reporting responsibilities thoroughly understand their policies.

Director of Operations

This individual must communicate all corporate policies to his or her employees and ensure that these policies and practices are being implemented and adhered to. This

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individual must be thoroughly familiar with all Environmental Data Collection Activities (EDCA's) which occur in the laboratory and is responsible for oversight of all aspects of laboratory operations. He/She sets the direction in the laboratory.

Laboratory Director

The Laboratory Director is responsible for streamlining the operation of the laboratory while following good laboratory practices and quality assurance guidelines. This individual is responsible for all aspects of laboratory operations, from sample preparation through final reporting of data. This includes ensuring that laboratory personnel are adequately trained, use proper techniques, follow SOP's, and adhere to quality assurance guidelines. This director interfaces with the Quality Assurance Officer to set and implement those guidelines. This individual has the final authority in approving a report for submission to a client, and is therefore responsible for the report's content. He or She is also responsible for SOP approval. The Laboratory Director is called upon to make decisions concerning usability of data in accordance with the data quality objectives of the project and good science.

Financial Manager/Administration

This individual is responsible for the confirmation and accurate billing of all services performed by the laboratory. He/she must ensure that the services performed are billed as documented to the client in the form of a quotation and that the terms and conditions of such an agreement are met by the laboratory. Additional responsibilities include approval of all purchases for expendable and capital items, disbursement and reconciliation of petty cash, preparation of fiscal year budget, tracking and reporting financial performance of operations, liaison for laboratory and corporate on financial position of the laboratory and management of administrative staff providing office and clerical support to the laboratory.

Quality Assurance Manager

The QA Manager provides technical support to and supervision of the quality assurance program. This officer has the responsibility of assuring that all EDCA's are adequately documented as well as all routinely used procedures which might impact data quality. He/she ensures that audits and reviews are being performed to assure adherence to quality control standards and that adequate follow-up actions are taken in response to any noted deficiencies. This officer plays a key role in ensuring that all personnel have access to any training or QA information needed to be knowledgeable in QA requirements, protocols, and technology. He/She is responsible for maintenance of records which document laboratory quality and serves as a liaison for quality assurance in dealings with clients and regulatory agencies. This Officer must review and approve all SOP's and implement and maintain the document control system. The Quality Assurance Manager reports to the Director of Operations, but also has a direct report responsibility to Corporate Management.

Sales and Marketing

These individuals are responsible for understanding and accurately communicating the Data Quality Objectives (DQO's) of a given project. They must ensure that the client's needs are being met through their communication with the laboratory and the client. This must be done as an ongoing process (ie. from initial client consultation through final report). Although responsible to the laboratory, the first priority must be to the client.

Field Services

These individuals are responsible for the development, planning and organization of the engineering department. They are responsible for acquiring the materials, equipment and trained personnel for specific engineering projects. They must ensure that the department has the capabilities to perform tasks required by a specific DQO and that the client's requests are completed with professionalism. They interact with the client, sales and marketing, department managers and group leaders. They are

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responsible for scheduling and coordinating sampling projects with the laboratory through project management. Their involvement begins with initial client consultation and ends with the final report.

Project Managers

These individuals interface with the client, sales and marketing, department managers and group leaders. They are responsible for knowing and understanding the DQO's specific to each project and accurately communicating this information to the laboratory. They must know the DQO-specific certification requirements and ensure that such requirements are met. Their involvement in each project starts at the initial planning stage and continues to the forward scheduling of work through the laboratory, monitoring its progress, review of the final report and, ultimately, reviewing the report with the client.

Reports Production

These individuals are responsible for the initial review, collation and drafting of final reports as well as the overall quality of the final product as it relates to the quality of typing, content and completeness. They must know and understand the DQO's of each project in order to confirm the laboratory's compliance with requirements. They interface directly with project management.

Systems Management

These individuals are responsible for the development and/or purchase of appropriate hardware and software to support efficient, cost-effective laboratory operations. They perform systems analyses for new or enhanced automation opportunities through specification development, vendor evaluation and system implementation, testing and validation. They are responsible for the training, support and maintenance of all automated data systems.

Sample Control

These individuals are responsible for the initial handling and accurate documentation of all samples received in the laboratory. They must ensure that each sample received meets the requirements of its DQO's as specified on the forward schedule and quotation and, when it does not, ensure immediate communication to project management. Sample custodians are responsible for inspection of coolers and samples (with appropriate custody seals and airbills), chain of custody, generation of job numbers, travellers and laboratory services requests. When applicable they are responsible for generation of subcontracting paperwork. They maintain all refrigerators and freezers used for sample storage.

Department Managers

These individuals must ensure that the analysts in their department understand and have the materials and capabilities to meet required DQO's for all given EDCA's. They have ultimate responsibility for the scheduling, planning and training in their department. They must ensure that all company policies and practices are understood by the analysts and that these practices are being implemented. These managers must approve all SOP's.

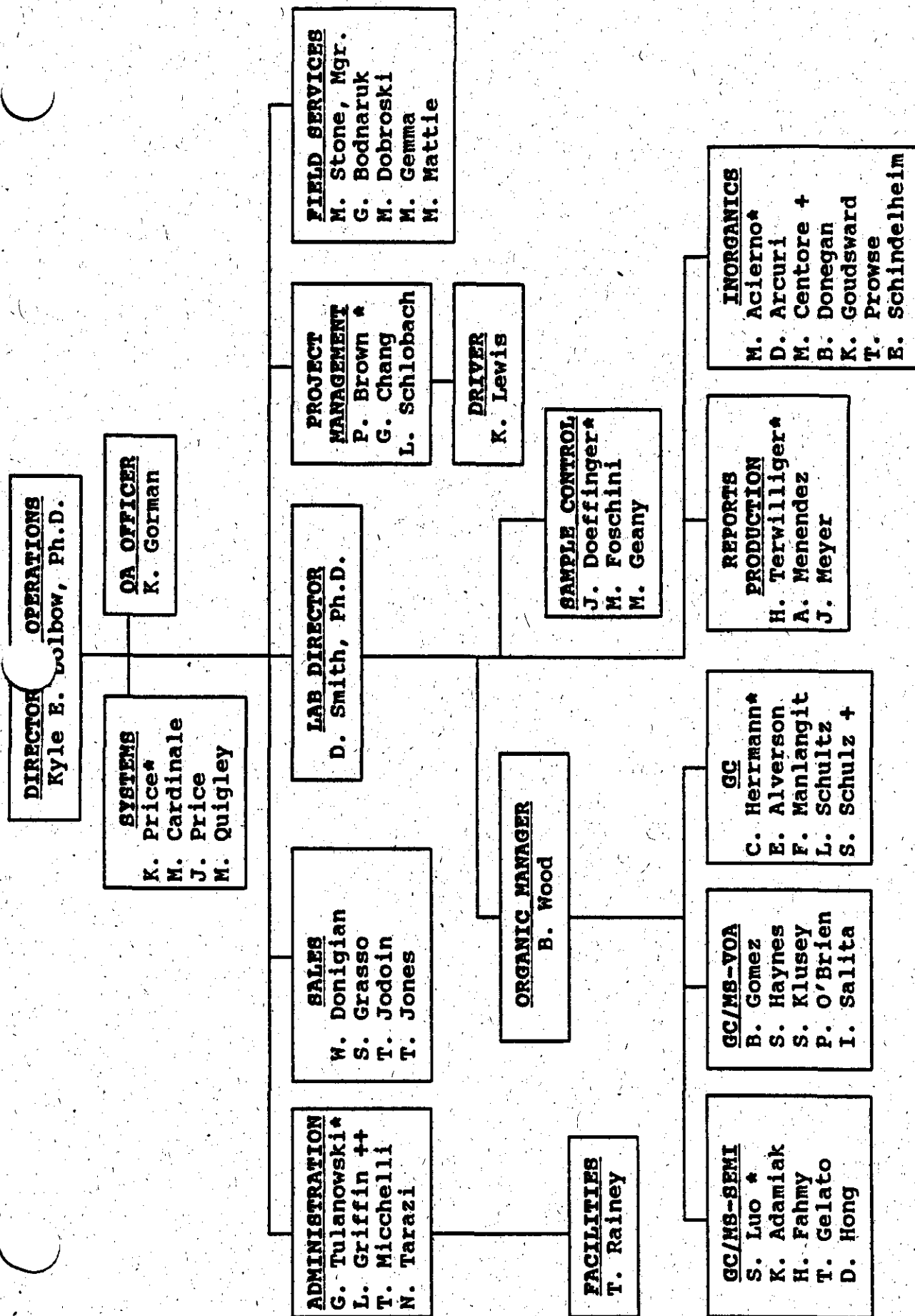
Group Leaders

These individuals represent first-line management and as such play a crucial role in laboratory operations. Their primary responsibility is ensuring data quality through implementation of SOP's based on DQO's communicated to them. They have first-line responsibility for report content. They are also responsible for communicating the required DQO's to their subordinates and ensuring that they are met. Training requirements, operational needs and general environmental conditions are all assessed and implemented at this level. These individuals represent the first SOP approval level within their department.

Individual Analysts

Each and every analyst is responsible for the quality of the data he or she produces. It is each person's responsibility to follow the requirements of the operation being performed, furthermore if the requirements are not understood, it is the individuals responsibility to ask for help or more information. Each individual must support the quality assurance program in principle and detail.

Figure 5-1 is an organizational chart for IEA - New Jersey.



* - GROUP LEADER ** - TEAM LEADER + - PART TIME ++ - TEMPORARY

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6.0 Facilities, Equipment, and Services

6.1 Facilities

IEA - New Jersey occupies a 14,000 square foot building of which approximately 70% is dedicated to the analytical laboratories. Separate laboratory areas are dedicated to GC and GC/MS instrumentation, volatile analysis, extractions for organic parameters, sample preparation for metals analysis, metals analysis by atomic absorption (GFAA) and inductively coupled plasma (ICP), wet chemistry and standards preparation.

The volatile analysis laboratory containing GC/MS instrumentation has a separate air handling system which maintains positive pressure at all times. The room is isolated from the rest of the laboratory, and the air that enters is filtered twice through two separate carbon filtering units. This design results in a contaminant-free environment for trace-level volatile analysis.

The laboratory has eleven (11) fume hoods strategically located for a total of over 66 linear feet to hood capacity. The instrumentation is powered by 110/220 volt circuits.

.2 Laboratory Instrumentation**Semivolatile Department****INSTRUMENT ID****DESCRIPTION****MSD**

Hewlett-Packard 5970 MSD equipped with
Hewlett-Packard 5890 GC
Hewlett-Packard 7673A Autosampler

MSG

Hewlett-Packard 5970 MSD equipped with
Hewlett-Packard 5890 Series GC
Hewlett-Packard 7673A Autosampler

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Volatile DepartmentINSTRUMENT IDDESCRIPTION

MSE

Hewlett-Packard 5995 GC/MS
Tekmar LSC-4000
Tekmar 4200
Tekmar Sample Heaters

MSF

Hewlett-Packard 5995
Tekmar LSC-2000
Tekmar ALS-2016

MSA

(Received June, 1993)

Hewlett-Packard 5972 MSD
Hewlett-Packard 5890 Series II GC
O.I. DPM-16
O.I. Model 4560
DOS Chemstation

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Gas Chromatography DepartmentINSTRUMENT IDDESCRIPTIONHP58901A
HP58901BHewlett-Packard 5890 GC equipped with dual
Electron-Capture Detectors
Hewlett-Packard 7673 Dual Tower Autosampler
Hewlett-Packard 35900 Dual Channel InterfaceHP58902A
HP58902BHewlett-Packard 5890 GC equipped with dual
Electron-Capture Detectors
Hewlett-Packard 7673 Dual Tower Autosampler
Hewlett-Packard 35900 Dual Channel InterfaceHP58903A
HP58903BHewlett-Packard 5890 GC equipped with
1 Flame Ionization Detector
1 Nitrogen-Phosphorous Detector
Hewlett-Packard 7673 Single Tower Autosampler
Hewlett-Packard 35900 Dual Channel InterfaceHP58904A
HP58904BHewlett-Packard 5890 Series II GC equipped with dual
Electron-Capture Detectors
Hewlett-Packard 7673 Dual Tower Autosampler
Hewlett-Packard 35900 Dual Channel InterfaceSCREENINGHewlett-Packard 5890 GC equipped with
Electron Capture Detector
Flame Ionization Detector
Hewlett-Packard 3396A Integrator
Hewlett-Packard 7673 Single Tower Autosampler

Date: 11/01/93
Page 19 of 46Sample Preparation DepartmentINSTRUMENT IDDESCRIPTION

GPC1	ABC 1002A Gel Permeation Chromatograph
GPC2	ABC 1000 Gel Permeation Chromatograph equipped with ABC UVD-1 Ultraviolet Detector ABC GR-1 Recorder
	3 Tekmar Sonic Disruptors equipped with 3/4" Q Horns

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Inorganics Department
Metals Laboratory

- 1 Thermo Jarrell Ash 61 Inductively Coupled Argon Plasma Emission Spectrometer equipped with Gateway 2000 Data System
- 2 Perkin-Elmer Zeeman 5100 Atomic Absorption Spectrophotometer equipped with
EPSON EX-800 Printer
Graphite Furnace
AS-60 Autosampler
EPSON Equity III Computer System, Gateway 2000 Data System
PE EDL Power Unit
- 1 Buck Cold Vapor Mercury Analyzer
- Leeman Labs PS200 Automated Mercury Analyzer

Inorganics Department
Wet Chemistry Laboratory

PE 1310 IR Spectrometer
HACH 2000 UV-VIS Spectrophotometer

Balances

Mettler AE 163 Analytical Balance
Mettler PE 3000 Top Loading Balance
Ohaus E 400 Top Loading Balances
Sartorius Top Loading Balance

Ovens/Furnaces

Thermolyne Type 18200 Muffle Furnace
VWR Model 1305U Oven
VWR Model 1320 Oven
VWR Model 1300U Oven
Precision Model 18EM Oven
Blue M Oven

Miscellaneous Laboratory Equipment

<u>Quantity</u>	<u>Description</u>
2	VWR Genie-2 Vortex Mixers
1	Amsco Autoclave
3	Tekmar Sonic Disruptors
1	Model 111-12 position N-EVAP
1	Burrell Wrist-Action Shaker
1	Precision 180 Water Bath
1	Boekel Water Bath
1	Blue-M Water Bath
2	Scientific Products IC-83 Incubators
1	Incubator Room
2	Lab Line Multi-Unit Extractors
2	Glass-Col Multi-Unit Extractors
2	Welsh Duo-Seal Model 1402 Vacuum Pumps
9	Fume Hoods
2	Portable Hoods
2	Captair Ductless Filtration Systems
5	Standard Freezer Units
1	VWR Model 1174 Refrigerated Recirculator
24	Corning 1-Step Liquid/Liquid Extractors
4	Supelco Liquid/Liquid Extractors
1	12-Port SPE Vacuum Manifold
2	VWR Model 1135 Constant Temperature Circulator
1	Sargent Welch Vacuum Pump
4	Millipore ZHE Extractors
8	Analytical Testing ZHE Extractors
2	Analytical Testing 8-Place Rotary Extractors
1	Analytical Testing 12-Place Rotary Extractor
1	Environmental Express 10-Place Rotary Extractor
50	Kontes Kuderna-Danish Evaporators
1	IEC HN-SII Centerfuge
1	VWR 1245PC Water Bath
2	Neslab CFT-33 Refrigerated Recirculators
1	Brown Walk-In Refrigerator

6.3 Services

The following is a list of the additional services offered by IEA - New Jersey:

- Data Validation
- Courier - Sample pick up and delivery
- Sample Container Preparation and Transport
- Hazardous Waste Assessment
- RCRA
- Effluent Monitoring
- Potable Water Analysis
- Real Estate Transfers
- Air Testing Capabilities
- Report Production
- Field Services (client requested)

Data Validation

IEA - New Jersey has performed data validation for several consulting and engineering firms that did not have an internal QA/AC department. We have performed validation services on New Jersey ECRA projects (Tier II reports) and Superfund remediation sites (CLP-Tier I reports). Mr. Kevin Gorman and Dr. Kyle Dolbow both have performed data validation on several projects.

Courier Service

IEA - New Jersey offers sample pick-up for local clients. This service was designed for customer convenience and has a small fee dependent upon the location of sample pick-up.

Sample Containers and Transport

IEA - New Jersey provides new, pre-labelled, preserved bottles for sample collection. Chain-of-custody documentation is initiated at the laboratory and included with the bottles. We can list up to 14 samples on a single form. This means less paperwork in the field and fewer mistakes.

There are two types of bottles that can be provided: Standard and I-Chem glassware (as used by the USEPA for CLP projects). Preservatives specified by the analytical methods are added to bottles before delivery to the client. The bottle labels identify both the parameter and preservative.

Bottles are delivered to the site in sample coolers chilled with blue ice, using Federal Express or the IEA - New Jersey courier service. Different size coolers are used depending on the number of containers. Each bottle is wrapped with protective "bubble wrap" prior to shipment. All coolers are sealed with custody tape. This is the same procedure used by the USEPA for Superfund site (CLP) projects. Coolers are returned to the laboratory by overnight courier, such as Federal Express, or the IEA - New Jersey courier service.

Hazardous Waste Assessments

CERCLA (Superfund)

Superfund site investigations, whether sponsored by Federal, State, or Principally Responsible Parties (PRP's) utilize the analytical guidelines established by the USEPA Contract Laboratory Program (CLP). Typical analytical requirements performed at Superfund sites include:

- Full Target Compound List (TCL's formerly known as the Hazardous Substance List-HSL's) air, soil and water.
- Tentatively Identified Compounds (TIC's) performed by GC/MS library search with analyst review.
- Site specific compounds, often times requiring some level of method development.
- Air analysis for volatile constituents potentially migrating from the site.

IEA - New Jersey has extensive experience providing these services to the New York State Department of Environmental Conservation, to EPA, served Fortune 500

companies in chemical, petroleum and pharmaceutical industries in New Jersey, as well as numerous other confidential industrial clients.

RCRA

The Resource Conservation and Recovery Act has a number of requirements for analytical testing. These requirements generally fall into two categories: analysis of waste to determine if the material is hazardous; and analysis of environmental samples (soils, water, air) to determine if hazardous constituents are present.

Analysis of waste material usually involves the Toxicity Characteristic Leaching Procedure Tests including:

- Volatile
- Semivolatile
- Pesticides
- Herbicides
- Metals

It is also common practice to test this material for:

- Priority Pollutants
- Appendix VIII constituents
- Other characteristics required by waste disposal companies

The 1986 RCRA Amendments also list a plan, phasing out certain hazardous materials from land disposal. The RCRA land ban program includes a number of analytical requirements. Some of these are:

- TCLP Solvent list (F001-F005 wastes)
- California List
- Appendix III

Analysis of environmental samples at RCRA facilities generally involves:

- Groundwater monitoring for RCRA indicators, Appendix IX, and specific constituent monitoring
- Soil analysis for Appendix IX or priority pollutants
- Air analysis for off-site migration of volatile organics

The guidelines for analytical testing for RCRA programs is provided in the SW846 Manual (Third Edition). IEA - New Jersey has reviewed the requirements of SW846 and has developed the analytical capabilities to comply with this document. We have carefully reviewed and are able to deliver all of the QA/QC described in SW846. Our understanding of the application and limitations of these methods helps us provide guidance to our clients. We are also able to clearly define, review, and manage our laboratory with the knowledge that we are providing quality data that meets the regulatory requirements.

Effluent Monitoring

NPDES

The Clean Water Act has certain requirements for effluent monitoring. This includes both the NPDES permit process for direct discharges and the Pretreatment program for effluents to POTW's. The NPDES permit generally involves priority pollutant analysis upon permit renewal (5 year) or on an annual basis, plus monthly reporting of "conventional pollutants". The Pretreatment program is based on industry specific categorical standards and the requirements of the POTW.

The analytical guidelines for these programs are described in 40 CFR Part 136, specifically, Methods 624/625 and 608 for organics and the 200 series for inorganics. The QA/QC requirements are described in these methods.

IEA - New Jersey has been providing effluent monitoring services since 1986. We provide sampling and analytical program design, sample collection and pick-up, and analysis using the appropriate methodologies and QA/QC.

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Potable Water Analysis**Safe Drinking Water Act (SDWA)**

The SDWA requires water purveyors test their supply at least once annually. Those supplying larger populations must test more frequently. There are a number of parameter groups that are tested under SDWA including the primary and secondary drinking water standards. These include:

Primary**Inorganics**

Arsenic, Barium, Cadmium,
Silver, Sodium, Chromium
Lead, Mercury, Selenium,
Fluoride, Nitrate
Surfactants, Zinc

Secondary**Inorganics**

Chloride, Color,
Copper, Iron,
Langelier Index,
Manganese, Odor, TDS, Sulfate,

Organics

Vinyl Chloride, Endrin, Lindane
Methoxychlor, Toxaphene
2,4-D, 2,4,5-TP (Silvex)

Organics

Vinyl Chloride

Biological

Total Coliform

Trihalomethanes and Volatile Organics**Radioactivity**

Gross alpha, beta, Radium 226, 228

The EPA promulgated amendments to the SDWA in 1986. The amendments include a "phase in" program for the analysis of volatile synthetic organic chemicals (SOC's), inorganic chemicals (IOC's), microbiological and radionuclide contaminants. The analytical protocols for the organic constituents are defined in the EPA 500 series methods.

IEA - New Jersey has provided drinking water analysis to many clients. We have also been contracted by the USEPA to perform a Special Analytical Services (SAS) project for the analysis of drinking water detection limit volatile organics. This required the use of capillary column purge and trap GC/MS using Method 524.2.

Real Estate Transfers

Many state environmental agencies have promulgated regulations requiring a site assessment prior to the sale of an industrial facility. The extent and complexity of the assessment varies due to different state regulatory requirements as well as site specific conditions. These transfers of property are governed by specific state environmental guidelines such as ISRA (NJ), Superlien (CT), and proposed regulations in Illinois.

IEA - New Jersey has provided laboratory services for numerous property transfers, particularly in New Jersey, under ECRA (now ISRA) regulations, and Connecticut under the Superlien Laws. The data generated from these projects have to withstand close scrutiny by state regulators. The data must also be of "courtroom quality" should there be any dispute over the findings.

We have the capability to test for specific chemicals and chemical groups as well as broad based chemical lists such as Priority Pollutants, RCRA characteristics, and the Target Compound List. We can provide rush turnaround time service for "hot" transactions as quickly as 24 hours for volatile organics. The most important aspect of this type of testing, however, is the data quality and its usability for the site assessment.

Air Testing Capabilities

IEA - New Jersey has been providing air testing services via subcontracting since the 1980's. We specialize in the analysis of volatile organic compounds for ambient air monitoring programs. We are also experienced in the analysis of other organic compounds, as well as inorganics.

Our experience includes several different sampling media such as Tenax tubes, Tedlar bags, SUMA Canisters, impinger solutions, and a variety of adsorptive traps. Existing air methods, for example T01 and T02 volatile organics, do not have the rigorous procedures and QA/QC detail. This requires that the laboratory have sufficient working knowledge and experience to provide quality, defensible data.

IEA - New Jersey has performed air testing for several clients, including the USEPA. We were contracted under the EPA's Special Analytical Services (SAS) program to provide trace analysis of volatile organics from SUMA canisters. We have conducted stack gas monitoring at a midwest steel mill, formaldehyde analysis on impinger solutions from a chemical manufacturing facility, soil vent gas testing from landfills in New Jersey, as well as numerous other projects.

As part of our air testing services, we provide consultation on sampling program design, including the selection of the most appropriate sampling media.

Our organization has investigated the adsorptive properties of many different traps and are able to pass this information, as well as our experience, to our customers. We are very proficient in the analysis of Tenax/adsorptive media traps using cryofocus GC/MS techniques. Using this knowledge, and the customers knowledge of site conditions, we can develop the most effective sampling plan.

We believe that our understanding and experience, combined with our familiarity with EPA-CLP QA/QC requirements, makes us uniquely qualified to provide air testing services.

7.0 Quality Assurance Objectives

The objectives of IEA are to supply precise, accurate data reports to clients which are representative of the sample supplied. All data reported are generated and calculated according to published methods which are recognized standards of the environmental laboratory industry. Thus, data reported by IEA are calculated and reported in units which are consistent with data produced by other organizations. IEA strives to ensure that each data report is 100% complete, containing all data elements and supporting documentation for the type of report requested by the client.

7.1 Quality Assurance Project Plans (QAPjP)

Each service provided by IEA must have associated with it a specific QAPjP. The QAPjP is developed to identify the types of monitoring systems are to be used throughout the project. Section 6.0 discusses each type of service provided. These plans are written by the Quality Assurance Officer and include the following sections:

- 1.0 Title Page, with provisions for approval signature
- 2.0 Table of Contents
- 3.0 Project Description
- 4.0 Project Organization and Responsibilities
- 5.0 QA Objectives for Measurement Data, in terms of precision, accuracy, completeness, and representativeness
- 6.0 Sampling Procedures
- 7.0 Sample Custody
- 8.0 Calibration Procedures and Frequency
- 9.0 Analytical Procedures
- 10.0 Data Analysis, Validation, and Reporting
- 11.0 Internal QC Checks and Frequency
- 12.0 Performance and System Audits and Frequency
- 13.0 Preventive Maintenance Procedures and Schedules
- 14.0 Specific routine procedures to be used to assess data precision, accuracy and completeness of specific measurement parameters involved
- 15.0 Corrective Action
- 16.0 Quality Assurance Reports to Management

2 Standard Operating Procedures

A standard format has been defined in IEA Document #QAS00200.NET and implemented for all IEA - New Jersey SOP's. A copy of this and all facility SOP's are available for review during a site visit.

SOP's are used throughout the laboratory to document the proper performance of various activities. All SOP's are document controlled to ensure outdated documents are not utilized. The following is a list of required SOPs for IEA - New Jersey:

- Sample Receipt and Logging
- Chain-of-Custody Procedures
- Sample Storage
- Security of Samples and Laboratory Facility
- Preventing Sample Contamination
- Purity of Standards and Standards Preparation Documentation
- Maintaining Laboratory Records and Logbooks
- Sample Analysis and Data Control Systems
- Sample Bottle and Glassware Cleaning Procedures
- Monitoring of Refrigerators, Freezers, and Ovens
- Monitoring of Laboratory Reagent Water Quality
- Technical Review of Data and Reports
- Sample Analysis, Data Handling and Reporting
- Instrument Preventative Maintenance
- Document Control System
- Corrective Action Process

8.0 Data Processing

8.1 Collection

Data is collected directly from the individual instruments into a central relational database (ORACLE) via an RS232 serial transmission. All wet chemistry parameters and organic extraction data are entered directly into the database.

8.2 Validation

Data in the database is validated by comparing each compound to the QC limits given in a particular method immediately upon sample analysis. After sample analysis for a particular job is complete, the analyst retrieves a hard copy of the data from the database. The data is reviewed for completeness and accuracy. The group leader then reviews the data against the project requirements to ensure that the data quality objectives have been met. Once the DQO's have been met, the group leader signs off on the fraction. The fraction is then given to reports production where each fraction is reviewed for completeness and a final report is generated.

8.3 Storage

Data is stored on the hard drive of the database and backed up daily onto DAT tapes. Hand written data is stored in either a bound notebook or in each project folder. Final reports are stored for 6 months on site, at which time they are shipped to an off site storage facility and kept for 5 years.

8.4 Data Reduction and Reporting

The data associated with each analysis are hardcopied for permanent storage either through the printing of computer files or through hand entry into bound laboratory notebooks. All notebook entries are dated and signed by the analyst. Standardized notebook and logbook requirements include the following:

- (01) Preprinted pages
- (02) Prenumbered pages
- (03) Bound logbooks
- (04) Document controlling of logbooks
- (05) Archival of old logbooks
- (06) Acceptance criteria in logbook
- (07) Making corrections
- (08) Secondary review of logbook entries

Notebook entries, or any other general laboratory records, must be made in blank ink.

Any logbook or notebook entries that are corrected are made by using a one-line strikeout in black ink. All corrections are signed and dated.

Data reduction includes all processes that change either the form of expression (i.e., the units of measure) or the quantity of data values (rounding). It often involves statistical and mathematical analysis of data and usually results in a reduced subset of the original data set. Data reduction is performed either manually by the analyst or by computer systems interfaced to the analytical instruments. Whenever such procedures are employed within the laboratory Network, mathematical procedures have been verified for accuracy of computation.

All data are subjected to a multilevel review. All data reports are reviewed by the group leader prior to release for final report generation. A cross section of data reports are reviewed by the Laboratory Director. All final data reports are reviewed by the department manager prior to release to the client. It is the responsibility of the Quality Assurance Manager to review a random sample of five percent of final reports prior to shipment.

Out-of-control conditions identified by the analyst, supervisor, manager or technical staff member are investigated, corrected and documented. Out-of-control conditions which are caused by the sample itself, are addressed in a project narrative in the final report.

All elements of the IEA - New Jersey Quality Assurance Program must be satisfied before a data report may be released to the client.

9.0 Data Quality Assessment

9.1 Instrument Calibration

Because of the critical relationship between instrument calibration, the accuracy of the analytical data generated, and specific method protocols that determine data quality, IEA maintains strict controls on the calibration procedures for the various types of analytical equipment. Each type of instrumentation is calibrated prior to sample analysis according to method criteria. Specific criteria for the instrument calibrations must be met before samples may be processed. Corrective action must be taken to remedy any out of control situations. Deviations from the stated criteria are strictly forbidden.

Hardcopy records of all instrument calibrations are maintained within the various departments. These records are reviewed by the department managers and/or supervisors and are audited semi-annually by the local Quality Assurance Manager and/or the Corporate Director of Quality Assurance.

9.2 Laboratory Quality Control Checks

The analytical process is continually checked and verified through the analysis of method blanks, instrument blanks, matrix spike blanks, matrix spikes, duplicate matrix spikes and sample duplicates. The department supervisor is responsible for the administration of the quality control checks within each department. The records associated with the administration of the quality control checks are reviewed as part of

the system and performance audits conducted by the Quality Assurance Manager. The data generated for each quality control sample are reviewed by the department supervisor, Laboratory Director or Manager.

All analytes are required to have concentrations of less than the Method Detection Limit (MDL) or CRQL in all method and instrument blanks. Analysis of samples should not proceed until this requirement is met. Noncompliance with respect to system blanks would result in data qualification, and this would be documented in the report to the client.

Blanks, Matrix spike blanks, Matrix spikes, Matrix spike duplicates and duplicates are prepared and analyzed for all organic analyses at a rate of one pair for every twenty (20) samples. The data generated for each pair of matrix spike duplicates are used to measure the precision and accuracy of the analysis. Acceptance criteria for the matrix spike duplicate pairs analyzed for the common organic analyses are listed in Tables 9-1 and 9-2 of this section.

Surrogate compounds are routinely used for the organic analyses. Compounds similar to the analytes of interest are added to each sample prior to initiation of the analytical process. The recovery of the surrogate(s) in the final analysis is used to evaluate the accuracy and precision of the process in each sample. Acceptable surrogate data must be obtained for each set of reported sample data. A database of surrogate recovery data for all samples is maintained. This database is reviewed periodically by the department supervisor, as well as by the Quality Assurance Manager. Current surrogate control limits for the common organic analyses are listed in Table 9-3 of this QA Program Plan.

Internal standards are employed for all GC/MS analyses. The internal standard areas are evaluated periodically to ensure the consistency of the injection techniques used throughout the laboratory.

Control charts are used as part of the overall QA program to determine if a procedure is both accurate and precise. Control limits are calculated from the data set; and any data points not within the limits is considered suspect.

The QA Officer generates control charts quarterly for all analyses. IEA generates three charts. The first is a chart of the % recovery of the continuing calibration check or standard. This can be used to determine the accuracy of the analysis. The second is a chart of the % recovery for the spike and spike duplicate which is also used to determine the accuracy. The third is a chart of the % difference between the duplicates (either spike or sample) and helps determine the precision. Table 9-4 presents the calculations used for precision and accuracy, along with the definitions of completeness, comparability and representativeness. IEA - NJ Document # QAS02000.NJ discusses the procedures for generating control charts, and is available during an on site visit.

Duplicate sample analyses are performed for the metals and inorganic parameters at a rate of one duplicate for every ten sample analyses. Warning and control limits have been established for each parameter. The specific limits have been included in the individual SOP's. Typical limits for the metals analyses are:

Analyte Concentration Range Limit	<u>Relative Percent Difference (RPD)</u>	
	Warning Limit	Control
0.03 - 0.20 mg/L	35%	65%
0.21 - 2.00 mg/L	15%	20%

Spiked sample analyses are performed for the metals and inorganic parameters at a rate of one spike for every ten sample analyses. Warning and control limits have been established for each parameter. The specific limits have been included in the individual SOPs. Typical limits for metals analyses are:

Analyte Concentration Range	Warning Limit % Recovery	Control Limit % Recovery
0.03 - 0.20 mg/L	$\pm 25\%$	$\pm 40\%$
0.21 - 2.00 mg/L	$\pm 15\%$	$\pm 20\%$

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Table 9-1: Spike Acceptance Criteria for Common Organic Analyses in Water

Semivolatiles by GC/MS

		<u>% Recovery</u>	<u>RPD</u>
BN	1,2,4-Trichlorobenzene	39 - 98	28
BN	1,4-Dichlorobenzene	36 - 97	28
BN	N-nitroso-di-n-propylamine	41 - 116	38
BN	Acenaphthalene	46 - 118	31
BN	2,4-Dinitrotoluene	24 - 96	38
BN	Pyrene	26 - 127	31
A	Phenol	12 - 110	42
A	2-Chlorophenol	27 - 123	40
A	4-Chloro-3-methylphenol	23 - 97	42
A	4-Nitrophenol	10 - 80	50
A	Pentachlorophenol	9 - 103	50

For acceptance, the majority of % recoveries and RPDs for each compound class (acid or base-neutral) in the duplicate spike pair must meet the % recoveries and RPDs listed above.

Volatiles by GC/MS

	<u>% Recovery</u>	<u>RPD</u>
1,1-Dichloroethane	61-145	14
Trichloroethene	71-120	14
Benzene	76-127	11
Toluene	76-125	13
Chlorobenzene	75-130	13

For acceptance, the majority of % recoveries and RPDs obtained for the duplicate spike pair must meet the % recoveries and RPDs listed above.

Pesticides by GC

	<u>% Recovery</u>	<u>RPD</u>
Lindane	56 - 123	15
Heptachlor	40 - 131	20
Aldrin	40 - 120	22
Dieldrin	52 - 126	18
Endrin	56 - 121	21
4,4'-DDT	38 - 127	27

For acceptance, the majority of % recoveries and RPDs obtained for the duplicate spike pair must meet the % recoveries and RPDs listed above.

Table 9-2: Spike Acceptance Criteria for Common Organic Analyses in Soil

Semivolatiles by GC/MS

	<u>% Recovery</u>	<u>RPD</u>
BN 1,2,4-Trichlorobenzene	38 - 107	23
BN 1,4-Dichlorobenzene	28 - 104	27
BN N-nitroso-di-n-propylamine	41 - 126	38
BN Acenaphthalene	31 - 137	19
BN 2,4-Dinitrotoluene	28 - 89	47
BN Pyrene	35 - 142	36
A Phenol	26 - 90	35
A 2-Chlorophenol	25 - 102	50
A 4-Chloro-3-methylphenol	26 - 103	33
A 4-Nitrophenol	11 - 114	50
A Pentachlorophenol	17 - 109	47

For acceptance, the majority of % recoveries and RPDs for each compound class (acid or base-neutral) in the duplicate spike pair must meet the % recoveries and RPDs listed above.

Volatiles by GC/MS

	<u>% Recovery</u>	<u>RPD</u>
1,1-Dichloroethane	59 - 172	22
Trichloroethene	62 - 137	24
Benzene	66 - 142	21
Toluene	59 - 139	21
Chlorobenzene	60 - 133	21

For acceptance, the majority of % recoveries and RPDs obtained for the duplicate spike pair must meet the % recoveries and RPDs listed above.

Pesticides by GC

	<u>% Recovery</u>	<u>RPD</u>
Lindane	46 - 127	50
Heptachlor	35 - 130	31
Aldrin	34 - 132	43
Dieldrin	31 - 134	38
Endrin	42 - 139	45
4,4'-DDT	23 - 134	50

For acceptance, the majority of % recoveries and RPDs obtained for the duplicate spike pair must meet the % recoveries and RPDs listed above.

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Table 9-3: Surrogate Control Limits**Semivolatiles by GC/MS**

—— % Recovery ——

	<u>Water</u>	<u>Soil</u>
Nitrobenzene-d5	35 - 114	23 - 123
2-Fluorobiphenyl	43 - 116	30 - 115
Terphenyl-d14	33 - 141	18 - 137
Phenol -d6	10 - 94	24 - 113
2-Fluorophenol	21 - 100	25 - 121
2,4,6-Tribromophenol	10 - 123	19 - 122
2-Chlorophenol-d4	33 - 110*	20 - 130*
1,2-Dichlorobenzene-d4	16 - 110*	20 - 130*

Volatiles by GC/MS

—— % Recovery ——

	<u>Water</u>	<u>Soil</u>
Toluene-d8	88 - 110	81 - 117
Bromofluorobenzene	86 - 115	74 - 121
1,2-Dichloroethane-d4	76 - 114	70 - 121

Pesticides by GC

—— % Recovery ——

	<u>Water</u>	<u>Soil</u>
Tetrachloro-m-xylene	60 - 150*	60 - 150*
Decachlorobiphenyl	60 - 150*	60 - 150*

*These control limits serve as warning limits not control limits per CLP. All other limits are control limits.

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9.4 Specific Quality Control Procedures for Data Quality Assessment

Data quality is assessed based on five main characteristics of the data which are:

Precision
Accuracy
Completeness
Representativeness
Comparability

Each of these characteristics is discussed below.

Precision

Precision is a measure of the reproducibility among replicate analyses. Data acceptance during normal processing is based on the specific control limits for the common analyses. Data obtained for all duplicates are evaluated monthly by the Quality Assurance Manager for developing trends. Relative Percent Difference (RPD) is used as the measure of precision between matrix spike duplicates. The formula utilized to calculate RPD is as follows:

Relative Percent Difference (RPD)

$$RPD = \frac{(MS - MSD)}{\text{Mean of } MS + MSD} \times 100$$

where MS = Percent Recovery for Matrix Spike
MSD = Percent Recovery for Matrix Spike Duplicate

Accuracy

Accuracy is a measure of the degree of difference between the analytically obtained value and the known or actual value for a sample. IEA assesses accuracy by determining the percent recovery (%R) for analytes contained by designated quality control samples. Data obtained for all laboratory spikes are evaluated monthly by the Quality Assurance Manager for developing trends.

$$\text{Percent Recovery} = \frac{\text{Analytical Value}}{\text{Spiked Value}} \times 100$$

Completeness

Completeness is a measure of the valid data obtained from an analysis expressed as the percentage of the total data that should have been obtained.

$$\% \text{ Completeness} = \frac{\text{Amount of Valid Data Obtained}}{\text{Total Amount of Valid Data Expected}} \times 100$$

IEA's objective for completeness is to be able to provide analytical data for 100% of samples received intact and have sufficient sample volume for conducting reanalysis if initial analysis does not meet QC acceptance criteria.

Representativeness

Representativeness of the analytical data is primarily a function of the sampling procedures and techniques employed in the field. As such, the sampling plan must be designed to provide representative samples to the laboratory. Once received at the laboratory, samples are homogenized as much as possible to yield representative data on the sample submitted for analysis.

Comparability:

The IEA objective for comparability is that all data be fully comparable with data from other Network laboratories. This is accomplished through use of the following practices:

- * Demonstrate traceability of standards to NIST or EPA sources.
- * Use of Standard and Approved methodologies
- * Standardized units of measure.
- * Standardized QC Acceptance Criteria.
- * Participation in interlaboratory studies to demonstrate laboratory performance.

10.0 Corrective Action

Corrective actions can be initiated at several operational levels, however, they always involve QA personnel. In each case, after an assessment of the situation, appropriate steps are taken to correct the problem. Depending on the severity of the problem, corrective actions may be taken at the analyst level, department level, or within the entire laboratory. IEA recognizes the importance of corrective action to maintain a high quality program. In this light, all data are reviewed for completeness, accuracy, and compliance with QC criteria both within the analytical laboratory by peer review and by the department supervisor or manager. IEA - New Jersey refers to all "corrective actions" as "problem documentation." The procedures for this operation can be found under the IEA SOP for Problem Documentation, Document # QAS00500.NJ and is available for review during an on site visit.

In general, there are three major types of corrective actions which may be initiated at IEA:

Sample Problems

Individual samples or matrix problems are usually handled within the analytical laboratory. Corrective actions may include complete reextraction, reparation, reanalysis, clean-up, dilutions or matrix modifications. All actions taken are documented with the analytical results.

QC Batch Problems

An entire batch of samples may require corrective action if QC criteria are not met. Department managers and QA staff are involved in the decisions for actions which include reanalysis, reextraction, etc. The QA department personnel review both sets of data where applicable to determine if the problems have been resolved.

Systematic Problems

Those problems of a procedural nature are handled by the laboratory managers and QA manager. For major operational changes, initiation of such are made only after approval by the QA manager and the Laboratory Director.

10.1 Performance and System Audits

Audits are another area that might warrant corrective action. Each type of audit conducted by IEA is discussed below.

Each quarter the overall performance of the laboratory staff is evaluated and compared to the performance criteria outlined in the quality assurance manual and the standard operating procedures. The local Quality Assurance Manager conducts a laboratory audit semi-annually to evaluate the performance of the laboratory staff and compares that performance to the requirements of the quality assurance program. The Corporate Director of Quality Assurance also conducts semi-annual system audits to provide the quarterly audit frequency. During this process, the records, standard operating procedures and adherence to those standard operating procedures are examined. The results of the audit process are summarized and issued to each department supervisor, Laboratory Director and/or Manager, Director of Operations and the Corporate QA Director.

Known intralaboratory performance samples are analyzed in the form of sample spikes, duplicates and duplicate sample spikes on a continuing basis. Two (2) such samples are processed for every twenty (20) production samples.

IEA participates in interlaboratory performance audits through the various state and federal certification programs. IEA is an active participant in the U.S. Environmental Protection Agency's Contract Laboratory Program (CLP). A list of IEA - New Jersey certifications, as well as those for our other five network laboratory operations, summarizes our analytical capabilities. IEA is in the process of implementing an internal blind PE program. The corporate quality assurance office will supply each QA Manager with performance evaluation samples which are run with government samples.

1.0 APPROVALS

The signature of the following individuals indicates that this Standard Operating Procedure (SOP) is complete and meets the requirements specified in Corporate Document # QAS00200.NET. In addition, it also signifies that the content meets the specifications of the referenced "Test Code"

Laboratory Director

Quality Assurance Manager

John F. Wood
Philip J. Herman

2.0 SCOPE AND APPLICATION

- 2.1 This method covers the determination of chemical oxygen demand in surface waters and domestic and industrial wastes. The applicable range of the method is 25 to 1500 mg/L.

3.0 SUMMARY OF METHOD

- 3.1 Samples, blanks and standards in sealed tubes are heated in a block digester in the presence of potassium dichromate at 150°C. After two hours, the tubes are removed from the digester, cooled, and measured spectrophotometrically.

3.2 High Level COD (150-1000 mg/l)

Organic and oxidizable inorganic compounds are oxidized by potassium dichromate at high temperature. During the oxidation process, Cr^{+6} is reduced to Cr^{+3} . The Cr^{+3} is then measured spectrophotometrically at 620 nm.

3.3 Ultralow and Low COD (0-50, 50-150 mg/l)

Organic and oxidizable inorganic compounds are oxidized by potassium dichromate at high temperature. During the oxidation process, Cr^{+6} is reduced to Cr^{+3} . The Cr^{+6} left behind is measured spectrophotometrically at 420 nm.

- 3.4 This procedure is based on EPA method 410.4.

4.0 INTERFERENCES

- 4.1 Chlorides are stoichiometrically oxidized by dichromate and will give high COD values. Sufficient mercuric sulfate is added to the reagents to complex up to 2,000 mg/L chlorides before it reacts with the dichromate.

5.0 SAFETY

- 5.1 Gloves and eye protection must be worn at all times when in the laboratory; lab coats should be worn.

6.0 SAMPLE CONTAINERS, COLLECTION AND PRESERVATION

6.1 Sample Containers

Sample containers may be of glass or plastic that are purchased precleaned from the manufacturer. Bottles may not be reused.

6.2 Sample Collection

Samples are collected in precleaned bottles and preserved with concentrated sulfuric acid to pH < 2.

6.3 Sample Preservation

Samples are preserved with concentrated sulfuric acid to a pH < 2 and cooled to 4°C.

6.4 Holding Times

Samples are analyzed within 28 days of sampling.

7.0 APPARATUS AND MATERIALS

7.1 COD digestion block

7.2 HACH COD Vials

7.2.1 0-50 mg/l - Ultralow range

7.2.2 50-150 mg/l - Low range

7.2.3 150-1000 mg/l - High range

7.3 HACH 2000 Spectrophotometer

7.4 Eppendorf pipette

8.0 REAGENTS AND STANDARD PREPARATION

8.1 Deionized water (DI)

8.2 Stock COD Solution

Carefully weigh out 0.8500 g of potassium hydrogen phthalate (KHP) on an analytical balance. Add ~800 ml DI to a 1 liter volumetric flask and add the KHP. Dilute volumetrically to 1 liter, and ensure all the KHP dissolves by inverting the flask at least 20 times. This constitutes 1,000 mg/L as COD.

8.3 COD Working Solution

Prepare standards in the range of interest (ultralow, low, or high) by diluting the stock solution. See section 9.1.

9.0 CALIBRATION

9.1 Calibration Standards

Dilute a known amount of stock standard into 100 ml DI in a volumetric flask. The following table gives the volumes and their final concentration:

<u>mL stock standard</u>	<u>Final Volume (mL)</u>	<u>mg/L COD</u>
0	100	0
2	100	20
3	100	30
4	100	40
5	100	50
7	100	70
10	100	100
12	100	120
15	100	150
30	100	300
60	100	600
80	100	800
Stock	-	1000

Standard Curve Preparation

All calibration standards are run in triplicate. An average and standard deviation is calculated for each point and a calibration curve is determined using a blank and 5 standards.

10.0 QUALITY CONTROL

10.1 Method Detection Limits

Method detection limits are calculated based on the 40 CFR chapter 1 definition of method detection limit. A minimum of seven replicate standards prepared at 3-5 times the Instrument Detection Limit (IDL). Method detection limits are calculated by multiplying the standard deviation of all replicate results by student t values listed in 40 CFR.

10.2 Practical quantitation limits for this method is the same as the method detection limit.

10.3 Method Blank

A method blank is run with every batch of samples.

10.4 Matrix Spike and Matrix Spike Duplicates

Matrix spikes and matrix spike duplicates are performed every 20 samples and are prepared by pipetting 1 mL of standard COD and 1 mL of sample into a vial.

10.5 Continuing Calibration Verification (CCV)

A continuing calibration verification is run with each batch of samples at a concentration near the low point of the calibration curve. The CCV must be within 15 % of the calibration curve, if not a new curve must be prepared.

11.0 SAMPLE PREPARATION AND INSTRUMENTATION PROCEDURES

11.1 Preheat the COD digester block to 150°C before use by switching the Timer/Manual switch to manual.

11.2 Using a 1 mL Eppendorf pipette, add 2 mL of DI, standard or sample to individually labelled vials. Note: if upon addition of sample, the COD reagent immediately turns green, dilute the sample volumetrically, then add 2 mL of the diluted sample to the vial. Tighten the screw cap and shake the vial to thoroughly mix.

11.3 Ensure the COD block has reached 150°C and when it has, insert vials into the block. Wait for ready light to turn on and when it does set the timer for 120 minutes (2 hours). Switch the Timer/Manual switch to timer and let samples digest.

11.4 After the 2 hours are up and the vials have cooled, read absorbances of standards and samples using a spectrophotometer.

11.4.1 For high level vials (150-1000 mg/l), set the wavelength to 620 nm and record absorbance directly off the spectrophotometer. Zero absorbance is set on the spectrophotometer with the blank. Read absorbances of samples and calculate mg/L against absorbances of standards.

11.4.2 For low and ultralow vials (0-150 mg/l), set the wavelength to 420 nm, read absorbances directly off the spectrophotometer and subtract these readings from the blank to give a corrected absorbance. Zero the instrument by leaving the cell container empty and press "ZERO." The instrument will display "WAIT" and after a few seconds, 0.00 will be displayed. Insert the blank vial into the instrument and record the absorbance.

12.0 CALCULATIONS

12.1 High Level COD

Absorbances are read directly from spectrophotometer and concentration is calculated from calibration curve.

12.2 Ultralow and Low COD

Sample and standard absorbances are subtracted from blank absorbance giving a corrected absorbance. This is then used to calculate concentration.

12.3 % Recovery

$$\frac{\text{Actual (mg/L)}}{\text{Theoretical (mg/L)}} \times 100 = \% \text{ Recovery}$$

12.2 Spike Recovery

$$\frac{(\text{Spiked sample} + \text{sample conc., mg/L}) - (\text{sample conc., mg/L})}{(\text{Spike added mg/L})} \times 100 = \% \text{ Recovery}$$

12.3 Relative Percent Difference:

$$\frac{|\text{Sample 1} - \text{Sample 2}|}{(\text{Avg sample})} \times 100 = \% \text{ RPD}$$

13.0 ACCEPTANCE OF DATA

13.1 Continuing Calibration Verification (CCV)

The CCV must be within 85% - 115% recovery of the initial calibration curve. If not, a new curve must be made up.

13.2 Matrix Spike and Matrix Spike Duplicate (MS, MSD)

The MS and MSD must be within 75% - 125% recovery. If not, then reanalysis may be required.

13.3 Duplicates

Duplicate analysis must be within $\pm 20\%$. If not, reanalysis may be required.

13.4 If both the % recovery for MS and MSD and the RPD for duplicates are not within limits, reanalysis is required. If only one of these parameters is out of limits, see the group leader or Quality Assurance Officer for action.

14.0 REPORTING OF RESULTS

14.1 All data is to be reviewed by the group leader before turning into Reports Production.

15.0 SUPPLEMENTAL DOCUMENTS

15.1 None.

16.0 REFERENCES

16.1 EPA Method No. 410.4

16.2 Standard Methods for the Examination of Water and Wastewater, 17 ed. Method #5220.

17.0 SUBSTANTIVE REVISIONS

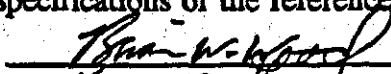
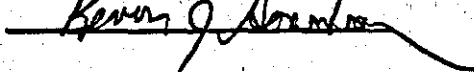
17.1 None at this time.

1.0 APPROVALS

The signature of the following individuals indicates that this SOP is complete and meets the requirements specified in corporate Document # QAS00200.NET. In addition, it also signifies that the content meets the specifications of the referenced "Test Code".

Laboratory Director

Quality Assurance Officer

2.0 SCOPE AND APPLICATION

- 2.1 This method is applicable to drinking, surface, and saline waters, domestic and industrial wastes.
- 2.2 The practical range of the determination is 10 mg/l to 20,000 mg/l.
- 2.3 USEPA method 160.1 is used by IEA - NJ.

3.0 SUMMARY OF METHOD

- 3.1 A well-mixed sample is filtered through a standard glass fiber filter. The filtrate is evaporated and dried to a constant weight at 180°C. The residue is then calculated as Total Dissolved or Filterable Solids.
- 3.2 Sample volume is 75 mls for this analysis, (the evaporating dishes can only hold this amount); The detection limit is 1.33 mg/l.
- 3.3 The holding time for analysis is 7 days after sample collection.

4.0 INTERFERENCES

- 4.1 Highly mineralized waters with a considerable calcium, magnesium, chloride, and/or sulfate content may be hygroscopic and require prolonged drying, proper desiccation, and rapid weighing.

5.0 SAFETY

- 5.1 Safety goggles/glasses are required, and in general, gloves and a lab coat should be worn.
- 5.2 Additional safety considerations are addressed in the IEA, Inc. - NJ Chemical Hygiene

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Plan. This plan should be thoroughly reviewed before any test is performed or any sample is handled.

6.0 SAMPLING CONTAINERS, COLLECTION AND PRESERVATION

6.1 Sample Containers

A non-preserved sample in resistant glass or plastic.

6.2 Sample Collection

Collect sample from source directly into bottles. Transport samples at 4°C.

6.3 Sample Preservation

Refrigeration or cooling at 4°C.

6.4 Holding Time

Samples must be analyzed within 7 days from collection.

7.0 APPARATUS AND MATERIALS

7.1 Glass microfiber filters (4.7 cm), Reeve Angel type 934-AH, Gelman type A/E or equivalent.

7.2 Suction flask, 500 ml.

7.3 Filter holder, membrane filter funnel or Gooch crucible adapter.

7.4 Evaporating dishes, porcelain, (100 ml volume).

7.5 Drying oven, 180°C $\pm$ 2°C, 103°C $\pm$ 2°C.

7.6 Vacuum Apparatus.

7.7 Desiccator.

7.8 Analytical balance, capable of weighing to 0.1 mg.

8.8 REAGENTS AND STANDARD PREPARATION

8.1 Deionized Water.

9.0 CALIBRATION

- 9.1 Analytical balances must be calibrated daily against class S weights. These procedures are covered under Document # QAS01000.NET.

10.0 QUALITY CONTROL

- 10.1 One blank, one sample replicate, one sample spike per batch of 20 samples.

- 10.1.2 Sample spike: Add 1 ml of KH_2PO_4 with 75 ml of sample. 1 ml of KH_2PO_4 = 781.0 mg/l.

11.0 SAMPLE PREPARATION AND INSTRUMENTAL PROCEDURES

- 11.1 Preparation of evaporating dishes: place the dishes in the oven (180°C) for at least one hour, then place in desiccator for at least one hour (or until a constant weight is obtained). Weigh immediately before use.

- 2 Assemble the filtering apparatus and begin suction. Shake the sample vigorously and rapidly transfer 75 ml to the funnel by means of a 100 ml graduated cylinder. Rinse cylinder into funnel.

- 11.3 Filter the sample through the glass fiber filter, rinse with distilled water and continue to apply vacuum for about 3 minutes after filtration is complete to remove as much water as possible.

- 11.4 Transfer the filtrate to a weighed evaporating dish and rinse filter flask with deionized water.

- 11.5 Place the evaporating dish in the 103° oven overnight. Then dry in oven at $180^\circ\text{C} \pm 2^\circ\text{C}$ for two hours.

- 11.6 Place dish in a desiccator for 1 hour. When the dish has cooled, weigh it and record the weight.

- 11.7 Reheat the dish in the 180°C oven for 1 hour and reweigh the dish until a constant weight can be obtained. Take final weight of dish. If the final weight change is more than 200 mg, the sample must be reanalyzed at a lower volume.

0 CALCULATIONS

- 12.1 Filterable residue, mg/l = $(A-B) \times 1,000,000$

C

Where

A = Weight of dried residue + dish (g)

B = Weight of dish (g)

C = Volume of sample used in ml.

13.0 DATA REVIEW

- 13.1 Final data is to be checked by a Team Leader or Group Leader who will turn in the report to report production.

14.0 REPORTING OF RESULTS

- 14.1 Data will be reported as mg/l Total Dissolved Solids. Any problems encountered will be discussed in the Case Narrative.

15.0 SUPPLEMENTAL DOCUMENTS

- 15.1 IEA Chemical Hygiene Plan.
- 15.2 Corporate SOP on SOP's, Document # QAS00200.NET.
- 15.3 Corporate SOP for Calibration of Lab Balances, Document # QAS01000.NET.

16.0 REFERENCES

- 16.1 Standard Methods for the Examination of Water and Wastewater, 17th Edition.
- 16.2 Methods for the Chemical Analysis for Water and Waste, 2nd Printing, 160.1-1. USEPA Method 160.1, (1983).

17.0 SUBSTANTIVE REVISIONS

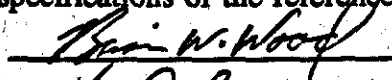
- 17.1 None at this time.

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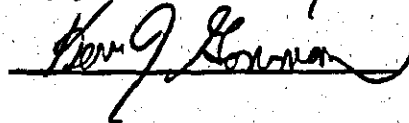
1.0 APPROVALS

The signature of the following individuals indicates that this SOP is complete and meets the requirements specified in corporate Document # QAS00200.NET. In addition, it also signifies that the content meets the specifications of the referenced "Test Code".

Laboratory Director



Quality Assurance Manager



2.0 SCOPE AND APPLICATION

- 2.1 Drinking, surface, and saline waters, domestic and industrial wastes are applicable to this method.

3.0 SUMMARY OF METHOD

- 3.1 A well mixed sample is filtered through a glass fiber filter, and the residue retained on the filter is dried to constant weight at 103-105°C.

- 3.2 Non-filterable residue is the solids which are retained by a glass fiber filter and dried to constant weight at 103-105°C.

- 3.3 This procedure is based on USEPA Method 160.2.

4.0 INTERFERENCES

- 4.1 Variables such as filtration apparatus, pre-washing, post-washing, filter material and drying temperature can affect TSS results.

- 4.2 Samples high in dissolved solids can be a positive interference. Correction for this involves selecting the right filtering apparatus so that the filter and any dissolved solids will be washed properly, thus, minimizes this problem.

5.0 SAFETY

- 5.1 Appropriate safety considerations are addressed in the IEA, Inc. - NJ Chemical Hygiene Plan. This plan should be thoroughly reviewed before any test is performed or any sample is handled.

6.0 SAMPLE CONTAINERS, COLLECTION AND PRESERVATION

- 6.1 Sampling Size - 100 ml sample

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6.2 Preservation - sample stored in plastic/NO preservative in refrigerator at 4°C.

6.3 Holding Time - 7 days from sample collection.

7.0 APPARATUS AND MATERIALS

7.1 Glass fiber filter discs (millipore AP-40, Reeves Angel 934-AH, Gleman A/E, or equivalent).

7.2 Filter support - filtering apparatus with reservoir and a coarse fritted disc as a filter support.

7.3 Suction flask, 500 ml.

7.4 Drying oven, 103-105°C.

7.5 Desiccator.

7.6 Analytical balance, capable of weighing to 0.1 mg.

7.7 Vacuum apparatus.

8.0 REAGENTS AND STANDARD PREPARATION

8.1 Deionized water.

9.0 CALIBRATION

9.1 The practical range of the determination is 4 mg/l to 20,000 mg/l.

10.0 QUALITY CONTROL

10.1 One blank and one duplicate must be run per matrix with every twenty samples.

10.2 A desiccator blank must be performed for every run.

10.3 Results are in mg/l.

11.0 SAMPLE PREPARATION AND INSTRUMENTAL PROCEDURES**11.1 Preparation of glass fiber filter disc.**

11.1.1 Place the glass fiber filter onto the bottom of a suitable Gooch crucible with wrinkled surface up.

11.1.2 While vacuum is applied, wash the disc with distilled water. Remove all traces of water by continuing to apply vacuum.

11.1.3 Remove filter from crucible and dry in oven at 103-105°C for one hour.

11.1.4 Transfer crucible to dessicator. Repeat the drying cycle until a constant weight is obtained (weight loss is less than 0.5 mg).

11.2 For a 4.7 cm diameter filter, filter 100 ml of sample. If weight of captured residue is less than 1.0 mg, the sample volume must be increased to provide at least 1.0 mg of residue.

11.3 Assemble the filtering apparatus and begin suction (wet filter first with distilled water).

11.4 Shake the sample vigorously and transfer 100 ml to the funnel by means of a graduated cylinder.

11.5 With suction on, wash the cylinder, filter, non-filterable residue and filter funnel wall with distilled water. Keep applying vacuum until all traces of water have been removed.

11.6 Carefully remove the filter from the filter support.

11.7 Remove crucible and filter from crucible adapter.

11.8 Dry crucible at least 1 hour at 103-105°C. Cool in desiccator and weigh. Repeat the drying cycle until a constant weight is obtained (weight loss is less than 0.5 mg).

12.0 CALCULATIONS

12.1 Non-filterable residue; mg/l =
$$\frac{(A-B) \times 1.000}{C}$$

where:

A = weight of filter (or filter and crucible) + residue

B = weight of filter (a filter and crucible) in mg

C = ml of sample filtered

13.0 ACCEPTANCE OF DATA

13.1 Final data is to be reviewed by the Group Leader before going to reports production.

14.0 REPORTING OF RESULTS

14.1 Data will be reported as Total Suspended Solids in mg/l.

15.0 SUPPLEMENTAL DOCUMENTS

15.1 Corporate SOP on SOP's, QAS00200.NET.

15.2 IEA Chemical Hygeine Plan

16.0 REFERENCES

16.1 USEPA Method 160.2, Total Non-Filterable Residue.

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

17.0 SUBSTANTIVE REVISIONS

17.1 None at this time.

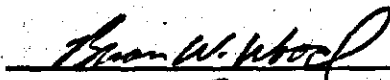
1.0 APPROVALS

The signature of the following individuals indicates that this SOP is complete and meets the requirements specified in corporate Document # QAS00200.NET. In addition, it also signifies that the content meets the specifications of the referenced "Test Code".

Laboratory Director



Organic Manager



Quality Assurance Manager



2.0 SCOPE AND APPLICATION

2.1 This method defines the specific steps for analyzing and determining the concentration of various organochlorine pesticides and polychlorinated biphenyls (PCBs) in environmental samples.

2 Refer to section 11.1 of this SOP for parameters.

3.0 SUMMARY OF METHOD

3.1 This method outlines the gas chromatographic procedure for the detection of organochlorine pesticides and polychlorinated biphenyls. Samples are extracted using the proper extraction technique. The extracts are then screened for dilution and analyzed by gas chromatography with an electron capture detector.

3.2 This SOP is based on SW846 Method 8080 (Organochlorine Pesticides and PCBs).

4.0 INTERFERENCES

4.1 Phthalate esters can interfere with pesticide determination; avoid any contact with plastic to best minimize this problem.

5.0 SAFETY

5.1 All analysts are required to wear safety glasses and gloves when working in the lab area or handling samples; lab coats are recommended.

Material Safety Data Sheets (MSDS) for all chemicals used in operations are present in

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the laboratory for immediate access. Specific MSDS's should be reviewed for a complete knowledge of the toxicity and precautions of each chemical.

6.0 SAMPLE CONTAINERS, COLLECTION AND PRESERVATION

6.1 Sample Containers

Aqueous samples are collected in 1.0 liter amber glass containers with Teflon-coated liners; soil samples are collected with 250 ml or 500 ml amber glass containers with Teflon-coated liners. Sample containers are never reused after sample analysis.

6.2 Sample Collection

Samples are collected as grab samples in the field.

A cooler with either ice or blue ice should be available at the sampling site.

6.3 Sample Preservation

Samples are preserved by cooling to 4 deg. C.

6.4 Holding Times

Aqueous samples must be extracted within 7 days and Soil samples must be extracted within 14 days from sampling. Samples must be analyzed within 40 days from date of extraction.

7.0 APPARATUS AND MATERIALS

7.1 Sample extracts are analyzed on a gas chromatograph (GC) equipped with an electron-capture detector (ECD), autosampler, data collection system and all other required accessories. The following columns are acceptable for quantitation and confirmation:

7.1.1 DB-608 30 meter
0.53mm ID 0.83um film thickness
J&W Scientific CAT #125-1730 or equivalent (ie. Restek RTX-35)

7.1.2 DB1701 30meter
0.53mm ID 1.0um film thickness
J&W Scientific CAT #125-0732 or equivalent (ie. Restek RTX1701)

7.1.3 DB5 30meter

0.53mm ID 1.5um film thickness

J&W Scientific CAT #125-5032 or equivalent (ie. Restek RTX-35)

7.1.4 Packed column: 3.0% SP2100 on 100/120 supelcoport
Supelco Inc. or equivalent

7.1.5 Packed column: 1.5% SP2250 1.95% SP2401 on 100/120
supelcoport
Supelco Inc. or equivalent

7.2 Various sizes of syringes, volumetric pipets, volumetric flasks, pipet bulbs

7.3 Sterile Borosilicate glass transfer pipets/transfer bulbs.

8.0 REAGENTS AND STANDARD PREPARATION

8.1 Solvents: Hexane, Acetone, Toluene, Isooctane (2,2,4-trimethylpentane) should be pesticide grade or equivalent.

2 Commercially prepared stock standards can be used if they are certified and pretested by the manufacturer.

8.3 All stock and working standards are stored in amber screw top bottles at 4 deg. C. Stock standards are replaced after one year and working standards are replaced after 6 months or earlier if necessary.

8.4 Calibration Stock Standards : See tables 1 and 2 for stock standard concentrations

8.5 Calibration Standards : Five (5) concentration levels are prepared through dilution of the calibration stock standard. One concentration level should be near but above the method detection limit (Refer to EPA Method #608). The remaining concentrations should correspond to the linear range of the instrument. See Table 1 and 2 for final component concentrations.

8.6 Instrument Breakdown Standard is prepared as a solution of p,p'DDT and Endrin to monitor breakdown in the injection port (see table 3).

9.0 CALIBRATION

9.1 Analyze the 5 levels of standards (Table 1 and 2) in the order of low to high. To verify the calibration, average response factor and relative standard deviations are calculated for each analyte.

9.1.1 Average Response Factors for single component pesticides are calculated by

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dividing the area response for a given analyte in each of the five calibration standards by the concentration of the standards and then calculating the average. A Relative Standard Deviation is calculated by dividing the standard deviation of the five response factors in the initial calibration by the average response factor.

An initial calibration is acceptable if the Relative Standard Deviation (RSD) is $\leq 20\%$.

9.1.2 Calculate the Average Response Factors for multicomponent analytes (ie Technical Chlordane, Toxaphene and Aroclors) by adding the total area of three (3) to five (5) of the major peaks in the multicomponent standard together. Divide the total area by the concentration of the standard. Use the same peaks in each calibration level and generate an average response factor and relative standard deviation as described above.

9.1.3 Retention Time Windows. The mean retention time from the initial calibration is determined and then windows are generated based on $\pm 2.0\%$ for packed column analysis and $\pm 0.3\%$ for capillary column analysis. A new window is generated after each calibration verification based on the same formulas except using the absolute retention time of each analyte in the calibration verification standard.

9.2 A calibration verification is performed every ten (10) samples to ensure the instrument calibration is still valid. The calibration verification consists of midlevel calibration standards for the single component analytes of interest. For Aroclors a representative Aroclor(s) is run to confirm linearity. This is usually a mixture of AR1016 and AR1260 unless otherwise specified. For Technical Chlordane and Toxaphene a five point calibration is included in the initial calibration. When a sample is analyzed that contains a pattern for either of these two compounds they are rerun after an acceptable midlevel calibration verification standard is run for these compounds. Isomers Alpha and Gamma Chlordane are included in the Individual Mix B. These may be used to identify possible Technical Chlordane or for quantitation of the isomers when pattern match for Technical Chlordane is not possible.

A continuing calibration verification is acceptable if the Relative Percent Difference (RPD) is $\leq 15\%$ for the quantitation column and $\leq 20\%$ for the confirmation column.

9.3 The calibration verification is acceptable if all of the following are true.

9.3.1 An instrument breakdown standard is to be analyzed as part of the first calibration verification for a sequence in which pesticide samples are to be analyzed. The breakdown of Endrin or p,p'DDT is $\leq 20\%$ based on the presence of Endrin Aldehyde and Endrin Ketone for Endrin or p,p'DDD and p,p'DDE for p,p'DDT.

If the breakdown does exceed 20% then corrective action must be taken prior to continuing the calibration verification.

% Breakdown for p,p'DDT

$$\frac{\text{DDD peak area} + \text{DDE peak area}}{\text{Total DDT peak area (DDT+DDD+DDE)}} \times 100$$

% Breakdown for Endrin

$$\frac{\text{Endrin aldehyde peak area} + \text{Endrin Ketone peak area}}{\text{Total Endrin peak area (Endrin+Endrin aldehyde+Endrin Ketone)}} \times 100$$

- 9.3.2 The midpoint calibration standard must have all compounds (+/-) 15% of their expected value. Because of the low concentration of pesticide standards injected on a GC/EC, column adsorption may be a problem when the GC has not been operated for a day. Therefore, the GC column should be primed by injecting a pesticide standard mixture approximately 20 times more concentrated than the midlevel standard.

If the midpoint calibration standard fails, then it can be rerun. If it fails two consecutive times, for any reason, a new calibration curve is to be run.

- 9.4 When analyzing samples, any extract that contains a target compound that exceeds the high level calibration must be diluted to within the calibration range.

- 9.5 Any sample chromatogram that displays a peak in the retention time window of a given analyte must be analyzed on another dissimilar column to confirm its presence. An analyte is determined to be present if a peak is present in the retention time window on both columns for that analyte. Multicomponent analytes are determined as above and by the matching of the pattern of peaks in the sample against that in the standards.

9.6 Calculations:

- 9.6.1 Quantitation of single-component target analytes: The calculation of targets is done by the software against the calibration curve using the following external standard calculation:

$$\text{Aqueous sample conc(ug/l)} = \frac{\text{area response of analyte}}{\text{average response factor for analyte in initial cal.}} \times \frac{V_f}{V_i} \times \text{dilution factor}$$

where:

Vf= final volume of the extract (ml)

Vi= initial volume of the sample (ml)

$$\text{Soil sample conc(ug/kg)} = \frac{\text{area response of analyte}}{\text{average response factor for analyte in initial cal.}} \times \frac{V_f}{V_i} \times DF \div \frac{\% \text{solid}}{100}$$

where:

Vf= final volume of the extract (ml)

Vi= initial volume of the sample (g)

9.6.2 Multicomponent analytes are quantitated in the same manner as above with the following exceptions:

1. Area response of the analyte is the total area of the three to five peaks used in the initial calibration.

2. Average response factor from the initial calibration is the average response factor of the three to five peaks chosen for quantitation in the initial calibration.

10.0 QUALITY CONTROL

10.1 A continuing calibration standard, breakdown standard is run at the beginning of an analytical sequence and then every 10 samples. The calibration standard must be within (+/-) 15% of expected value versus the curve; and breakdown must be $\leq 20\%$ each for Endrin and p,p'DDT.

10.2 Matrix spike (MS) and matrix spike duplicates (MSDs) are analyzed within every set of 20 samples or less. Recoveries must be within the limits listed in Section 13.4 of this SOP. If these criteria are not met, but the blank spike data meet all of the recovery criteria, then the MS/MSD are documented as having matrix interferences. If the blank spike fails, check for instrument and/or column related problems and reanalyze the spikes. If the problem is corrected, the samples are reanalyzed. If these spikes still fail the sample batch is reextracted.

10.3 A method blank must be analyzed with each extracted batch of samples. This should have no target compounds above PQL's.

10.4 Surrogates must fall within retention time windows. The %-recovery in samples, spikes, blanks is advisory from 60-150% for TCX and 20-150% for DBC in soils samples and 60-150% for TCX and 24-154% in aqueous samples.

11.0 INSTRUMENTAL PROCEDURES

11.1 Two HP5890 GC with dual EC's and 7673 Twin Tower Autosampler are currently being used to run Pesticides/PCBs under this SOP. Instruments, columns and conditions are as follows:

GC-HP58901A	Front Injection port:	30 meter DB-1701 (See Section 7.1)
	Column flow:	5.0 ml/min He
	Detector Make-up Flow:	55 ml/min P-5
	Injector Temperature:	205°C
	Detector Temperature:	325°C

GC-HP58901B	Back Injection Port	RTX-35 (See Section 7.1)
	Column Flow:	5 ml/min He
	Detector Make-up Flow:	55 ml/min P-5
	Injector Temperature:	205°C
	Detector Temperature:	325°C

Temperature Programming:

Initial Temperature: 170 deg. C Hold 1.0 min.

Initial Ramp: 3 degrees/min to 270 deg. C, Hold 1.67 min.

Total Analysis Time = 36 minutes.

GC-HP58902A	Front Injection Port:	Glass packed column containing 3.0% Sp2100 on 100/120 supelcoport
	Column flow:	45 mls/min P-5
	Injection Temperature:	250°C
	Detector Temperature:	325°C

GC-HP58902B	Back Injection Port:	Glass packed column containing 1.5% SP2250/1.95% SP2401 on 100/120 supelcoport
	Column Flow:	60ml/min P-5
	Injection Temperature:	250°C
	Detector Temperature:	325°C

Temperature Programming: Isothermal at 205°C for 40.0 minutes

11.2 Data System:

HP1000 Analytical Data System with A-400 CPU, running RTE-A Operating System and HP3350A Laboratory Automation System Software for data collection and integration. This data is exported to Automated Data Acquisition Manager (ADAM) for calculation and reporting.

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12.0 CALCULATIONS

See Section 9.6 of this document.

12.1.0 Calculation of % Difference:

The following formula is used to calculate % difference in the calculated versus expected values of standards.

$$\% \text{ Difference} = \frac{|\text{Calculated conc.} - \text{expected conc.}|}{\text{Expected conc.}} \times 100$$

12.1.1 Calculation of spike and QC check recoveries:

The following calculation is used for spiked sample recoveries.

$$\% \text{ Recoveries} = \frac{\text{Amount recovered}}{\text{Amount added}} \times 100\%$$

13.0 ACCEPTANCE OF DATA

13.1 Calibration Check Standard.

Verification of the calibration curve with the midlevel calibration standard is obtained if the calculated concentration of all the compounds is (+/-) 15% of the expected value. This is performed every 10 samples

13.2 Breakdown Check.

Instrument Breakdown of DDT and Endrin is considered under control if the % Breakdown of each analyte is $\leq 20\%$.

13.3 Method Blanks.

Method blanks are extracted with every batch of up to 20 samples to ensure that there is no contamination from the extraction process. The method blanks shall be free of any target compounds above quantitation limits and shall not contain any unusual interferences.

13.4 Matrix Spikes and Matrix Spike Duplicates

MS and MSD are extracted with every batch of up to 20 samples to verify extraction efficiencies. Acceptance criteria are listed below

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<u>PARAMETER</u>	<u>AQUEOUS RANGE FOR P (%)</u>	<u>SOIL RANGE FOR P(%)</u>
Aldrin	40-120	34-132
gamma-BHC	56-123	46-127
4,4'-DDT	38-127	23-134
Dieldrin	52-126	31-134
Endrin	56-121	42-139
Heptachlor	40-131	35-130

where:

$P = \% \text{ Recovery}$

14.0 REPORTING OF RESULTS

14.1 Three different levels of reports are provided for this method:

DATA SUMMARY

This deliverables package includes data for:

- (1) Analytical results for samples and the method blank.

REDUCED DELIVERABLES

In addition to analytical results for samples, this deliverable package includes data for:

- (1) Blank Analysis
- (2) Matrix spike/Matrix spike duplicate
- (3) Surrogate recoveries
- (4) Complete sampling chain-of-custody documentation
- (5) Retention time window for each standard in the initial calibration and continuing calibration.
- (6) Average response factor for each standard in the initial calibration.
- (7) Retention time shift and analytical sequence forms.

REGULATORY FORMATE In addition to the information contained in the reduced deliverables package, this deliverables package includes standards, samples and QC samples, all chromatograms and quatitation pages from the instrument data system.

15.0 SUPPLEMENTAL DOCUMENTS

15.1 SOP for Pesticides/PCB in Solid Extraction by Method 8080.

15.2 SOP for Pesticides/PCB in Aqueous Extraction by Method 8080.

16.0 REFERENCES

16.1 "Method 8080 - Organochlorine Pesticides and PCBs", EPA SW846, 3RD Edition.

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17.0 SUBSTANTIVE REVISIONS

17.1 None at this time.

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**TABLE 1 - Single Component
Calibration Concentrations, ug/mL**

Analyte	Stock	LEVEL1	LEVEL2	LEVEL3	LEVEL4	LEVEL5
Aldrin	0.5	0.005	0.010	0.020	0.040	0.080
Dieldrin	1.0	0.010	0.020	0.040	0.080	0.160
p,p'DDT	1.0	0.010	0.020	0.040	0.080	0.160
p,p'DDE	1.0	0.010	0.020	0.040	0.080	0.160
p,p'DDD	1.0	0.010	0.020	0.040	0.080	0.160
Endosulfan I	0.50	0.005	0.010	0.020	0.040	0.080
Endosulfan II	1.0	0.010	0.020	0.040	0.080	0.160
Endosulfan Sulfate	1.0	0.010	0.020	0.040	0.080	0.160
Endrin	1.0	0.010	0.020	0.040	0.080	0.160
Endrin Aldehyde	1.0	0.010	0.020	0.040	0.080	0.160
Heptachlor	0.50	0.005	0.010	0.020	0.040	0.080
Heptachlor Epoxide	0.50	0.005	0.010	0.020	0.040	0.080
alpha-BHC	0.50	0.005	0.010	0.020	0.040	0.080
beta-BHC	0.50	0.005	0.010	0.020	0.040	0.080
gamma-BHC	0.50	0.005	0.010	0.020	0.040	0.080
delta-BHC	0.50	0.005	0.010	0.020	0.040	0.080
Methoxychlor	5.0	0.050	0.100	0.200	0.400	0.800
Endrin Ketone	1.0	0.010	0.020	0.040	0.080	0.16
TCX	200	0.020	0.040	0.080	0.160	0.320
DBC	200	0.020	0.040	0.080	.160	.320
A-Chlordane	0.50	0.005	0.010	0.020	0.040	0.080
G-Chlordane	0.50	0.005	0.010	0.020	0.040	0.080
Chlorobenzilate	10.0	0.050	0.100	0.200	0.300	0.400
Kepone	5.0	0.025	0.050	0.100	0.150	0.200
Isodrin	5.0	0.025	0.050	0.100	0.150	0.200
Diallate	10.0	0.050	0.100	0.200	0.300	0.400

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**TABLE 2 - Multi-Component
Concentration, ug/mL**

Multi-Component	Stock	LEVEL1	LEVEL2	LEVEL3	LEVEL4	LEVEL5
AR 1660	1.0	0.10	0.25	0.60	1.50	4.0
AR 1221	1.0	0.10	0.25	0.60	1.50	4.0
AR 1232	1.0	0.10	0.25	0.60	1.50	4.0
AR 1242	1.0	0.10	0.25	0.60	1.50	4.0
AR 1248	1.0	0.10	0.25	0.60	1.50	4.0
AR 1254	1.0	0.10	0.25	0.60	1.50	4.0
Technical Chlordane	10.0	0.20	0.40	0.60	0.80	1.0
Toxaphene	10.0	0.25	0.60	1.5	4.0	10.0

TABLE 3 - Breakdown Std Conc., ug/mL

Compound	Stock (solution)	Standard (working)
Endrin	50.0	0.050
p,p'DDT	100	0.100

AR300144



IEA
An Aquarion Company

200 Monroe Turnpike
Monroe, Connecticut 06468

Phone 203-261-4458
Fax 203-268-5346

STANDARD OPERATING PROCEDURE

ANALYSIS OF AMMONIA (Method 350.1)

IN WATER

WC:070792:0

So.
Florida
205-845-1730

Schaumburg,
Illinois
708-705-0740

N. Dolema,
Massachusetts
617-272-8212

Whippany,
New Jersey
201-428-8181

Research Triangle Park,
North Carolina
919-577-0090

Essex Junction,
Vermont
802-878-5138

AR300145

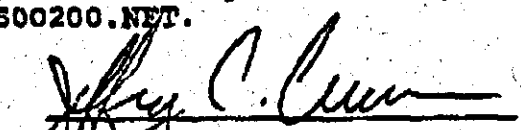
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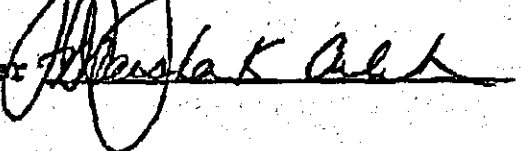
- 1.0 Approvals**
- 2.0 Scope and Application**
- 3.0 Summary of Method**
- 4.0 Interferences**
- 5.0 Safety**
- 6.0 Sample Preservation and Storage**
- 7.0 Apparatus and Materials**
- 8.0 Reagents and Standard Preparation**
- 9.0 Calibration**
- 10.0 Quality Control**
- 11.0 Sample Preparation and Procedure**
- 12.0 Calculations**
- 13.0 Acceptance of Data**
- 14.0 Reporting of Results**
- 15.0 Supplemental Documents**
- 16.0 References**
- 17.0 Substantive Revisions**
- Appendix I: Forms**
- Figure 1: System Schematic**
- Figure 2: Ammonia Development Order**

WC:070792:0

Ammonia by Automated Phenate Method**1.0 APPROVALS**

The signature of the following individuals indicates that this SOP is complete and meets the requirements specified in corporate Document #QAS00200.NET.

Laboratory Director 

Quality Assurance Manager 

2.0 SCOPE AND APPLICATION

- 2.1 This ammonia method is applicable to drinking, surface, and saline waters, and domestic and industrial wastes.
- 2.2 This method has a practical quantitation limit (PQL) of 0.04 mg/L and is suitable for concentration ranges of 0.04 to 4.00 mg/L.
- 2.3 This SOP is based on EPA method 350.1.
- 2.4 A preliminary distillation step is required for NPDES work to remove interferences. The distillation procedure is contained in document WC:021690.

3.0 SUMMARY OF METHOD

- 3.1 Alkaline phenol and hypochlorite react with ammonia to form indophenol blue that is proportional to the ammonia concentration. The blue color formed is intensified with sodium nitroprusside.

4.0 INTERFERENCES

- 4.1 Calcium and magnesium ions may be present in concentration sufficient to cause precipitation problems during analysis.
- 4.2 Sample turbidity and color may interfere with this method. Turbidity may be removed by filtration prior to analysis. Sample color that absorbs in the photometric range used will also interfere.

WC:070792:0

5.0 SAFETY

- 5.1 At a minimum, wear PVC gloves, a lab coat and eye protection when handling samples and reagents.
- 5.2 Always refer to your IEA Employee Safety Handbook prior to performing laboratory operations.

6.0 SAMPLE PRESERVATION AND STORAGE

- 6.1 Samples are to be collected in either a polyethylene or glass bottle and stored preserved with H_2SO_4 (pH<2) at 4 C until the time of analysis.
- 6.2 Samples are to be analyzed within 28 days of collection or within 26 days of receipt for NYSDEC protocol.

7.0 APPARATUS AND MATERIALS

- 7.1 Lachat QuikChem Analyzer; consisting of an autosampler, proportioning pump, injection module, IBM PC, chloride reaction board, colorimeter, and a chart recorder.
- 7.2 Pipets.
- 7.3 Volumetric flasks; 100, 250 and 1000 mL.
- 7.4 Vacuum filter apparatus
- 7.5 Membrane filters; 0.45 um.

8.0 REAGENTS AND STANDARD PREPARATION

- 8.1 All chemicals are to conform to the specifications set by the Reagent Chemical Committee of the American Chemical Society. Laboratory prepared "nano" water is used whenever reagent water is required.
- 8.2 Sodium phenolate: Dissolve 83 g of crystalline phenol in 600 mL of reagent water. While stirring, slowly add 32 g of sodium hydroxide and dilute to 1 liter.
- 8.3 Sodium hypochlorite: Dilute 250 mL of household bleach to 500 mL with reagent water. Degas with helium daily.

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- 8.4 Buffer: Dissolve 50.0 g disodium ethylenediamine tetraacetate and 5.5 g of sodium hydroxide in 900 mL of reagent water. Dilute to 1 liter.
- 8.5 Sodium nitroprusside: Dissolve 3.50 g of sodium nitroprusside in 1 liter of reagent water.
- 8.6 Stock ammonia standard (1000 mg as N/L): Dissolve 3.819 g of dried ammonia chloride in 800 mL of reagent water and dilute to 1 liter with reagent water.
- 8.7 The working standards are prepared as follows:
- 40.0 mg/L standard; Dilute 4 mL of stock to 100 mL.
 - 10.0 mg/L standard; Dilute 5 mL of stock to 500 mL.
 - 4.00 mg/L standard; Dilute 25 mL of 40 mg/L std to 250 mL.
 - 1.60 mg/L standard; Dilute 10 mL of 40 mg/L std to 250 mL.
 - 1.00 mg/L standard; Dilute 25 mL of 10 mg/L std to 250 mL.
 - 0.40 mg/L standard; Dilute 10 mL of 10 mg/L std to 250 mL.
 - 0.10 mg/L standard; Dilute 1 mL of 10 mg/L std to 100 mL.
 - 0.04 mg/L standard; Dilute 1 mL of 10 mg/L std to 250 mL.

Working standards may be stored at 4 C for up to one week.

- 8.8 Laboratory control standard: Prepared following the independent source suppliers instructions.
- 8.9 Helium (for degassing reagents).

9.0 CALIBRATION

- 9.1 The following instrument method settings are required before starting the calibration procedure (these settings are stored in the ammonia method file);

Pump speed = 35
Cycle period = 40 s
Load period = 15 s
Injection period = 25 s
Inject to start of peak period = 22 s
Inject to end of peak period = 58 s

9.2 Instrument Tuning

- 9.2.1 Set the Gain to 150 and adjust the baseline on the colorimeter to an LED readout of -0.200. The baseline should be stable before continuing.

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9.2.2 The high standard (4.00 mg/L) is manually injected and the GAIN adjusted in such a manner that the peak height is the size of the background analytical window.

9.2.3 The high standard manual injection is repeated to verify the peak height criteria.

9.3 A standard curve is prepared from data consisting of six working standard solutions (4.00, 1.60, 1.00, 0.40, 0.10, 0.04 mg/L) and a reagent blank. The calibration standards are placed in descending order into the autosampler tray and run.

9.4 The curve is run and each must pass a correlation coefficient of 0.995. The acceptance criteria is programmed into the PC and the calibration statistics are printed at the completion of the analytical run.

9.5 A continuing calibration verification (CCV) or check standard (1.00 mg/L) is run every tenth injection and at the end of the analytical run. Each CCV must meet a $\pm 15\%$ of the true value criteria to continue the analysis. A continuing calibration blank (CCB) is run following every CCV. The CCB must concentration must be less than the PQL (0.04 mg/L).

10.0 QUALITY CONTROL

10.1 The reporting or practical quantitation limit (PQL) for ammonia in water is 0.04 mg/L. The average relative percent difference for ammonia in 1991 was 2.86%, the average spike recovery was 97.8% over the same period of time. The LCS percent recoveries averaged 101.6% for 1991.

10.2 The working range for this ammonia procedure is 0.04 to 4.00 mg/L without dilution.

10.3 All stock solutions and standard preparations are logged and coded. All solutions are labeled with the following: analyte, concentration, analyst's initials, date prepared, and expiration date.

10.4 All chemicals will conform to minimum specifications set by the Reagent Chemicals Committee of the American Chemical Society. All chemical inventories are used on a first-in, first-out basis.

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- 10.5 A prep blank (reagent water) is to be analyzed with every batch of twenty or fewer samples. If a prep blank's concentration exceeds the PQL, all samples that were prepared with the prep blank and have a concentration greater than the PQL and less than 10 times the concentration of the prep blank must be rerun.
- 10.6 One laboratory control sample (LCS) is to be analyzed with each batch of twenty or fewer samples. LCS control limits are $\pm 15\%$ of the known concentration.
- NOTE: Should the LCS fail, the source of the problem must be corrected and an acceptable LCS run before continuing with the analysis of samples.
- 10.7 One sample duplicate must be analyzed from each batch of twenty or fewer samples. Samples identified as field blanks are not to be used for duplicate analysis. The relative percent difference (RPD) between the sample and duplicate must be less than or equal to 20.0 (see section 12.3 for the RPD calculation). No RPD criteria is applied if both results are less than five times the PQL. No RPD criteria is applied if one result is lower than five times the PQL and one above, if the difference between the two results is no more than the PQL.
- 10.8 A sample spike is performed on every batch of twenty or fewer samples (see 12.4 for spike recovery calc.) Samples identified as field blanks may not be used for sample spike analysis. The acceptable spike recovery range is 75 to 125%, if the sample concentration is greater than 4 times the spiking level, no criteria is placed on the spike.
- 10.9 If both the spike and duplicate fail to meet criteria, all associated samples must be reanalyzed. If only the sample duplicate or spike fails, a matrix interference is suspected and the failed QC is reanalyzed to confirm the results.
- 10.10 The batch size described for the PB, LCS, spike, and duplicate is for SOP protocol. SW-846 protocol projects require a spike, a spike duplicate, and a duplicate every ten samples.
- 10.11 Batch size and control limit requirements may be superseded or modified by a clients quality assurance plan for a specific project, check with supervisor about specific projects.
- 10.12 Corrective Action Reports (CAR) must be filed if any problems are associated with the analysis or any deviation

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from this SOP occurs (see appendix I).

11.0 SAMPLE PREPARATION AND PROCEDURE

- 11.1 All samples to be analyzed must be signed out of sample control on the chain-of-custody (COC) for each job.
- 11.2 If particulate matter is present, the sample must be filtered through a 0.45 um membrane filter prior to analysis.
- 11.3 System Operation:
 - 11.3.1 Turn on the PC, load the ammonia method file and enter the sample IDs and dilution factors.
 - 11.3.2 Inspect modules for proper connections. Turn on power to all modules, including the strip chart recorder. See figure 1 for the system schematic.
 - 11.3.3 Place reagent feed lines into proper containers. Raise tension levers on the pump tube cassettes.
 - 11.3.4 See section 9.0 for tuning and calibration.
 - 11.3.5 Load sample tray with samples and associated QC.
- 11.4 Rerun all samples with concentration over the high standard (4.00 mg/L) at a dilution. Dilutions are prepared using volumetric glassware.
- 11.5 Instrument Shut Down:
 - 11.5.1 At the end of the run, place all feedlines in water, flush the system and pump dry.
 - 11.5.2 Turn off pump, all modules, and releases levers on the tube cassettes.
- 11.6 Preventive Maintenance:
 - 11.6.1 The pump tubes, o-rings, and chloride reaction board tubing are checked daily and replaced as required, based on signs of physical wear.
 - 11.6.2 The manifold is rinsed with reagent water before and after use.
 - 11.6.3 Data files are purged from the hard disk and archived at least quarterly onto floppy disks.

WC:070792:0

2.0 CALCULATIONS

- 12.1 Dilution factors are entered into the PC when sample IDs for each tray are entered. The output from the instrument is in mg/L. The results are then entered into the lab LIMS system.
- 12.2 Manual ammonia calculation can only be done by using the linear regression of the daily curve generated and using the PC program source code to print the integration data.
- 12.3 The relative percent difference is calculated:

$$RPD = \frac{(S - D)}{(S + D)/2} \times 100$$

where: S = Original sample result
D = Duplicate sample result

- 12.4 The matrix spike recovery is calculated:

$$\text{Percent Recovery} = \frac{(SSR - SR) \times 100}{SA}$$

where: SSR = Spiked sample result (mg/L)
SR = Sample result (mg/L)
SA = Spike added (mg/L)

13.0 ACCEPTANCE OF DATA

- 13.1 The technical data review includes verifying that samples analyzed were preserved and that holding times were met. All quality control measures are checked to insure that all QC criteria described in section 10.0 were met. The raw data is examined for completeness including a secondary signoff. Results entered into LIMS are checked for transcription error against the raw data.

14.0 REPORTING OF RESULTS

- 14.1 Batch quality control is reported on the inorganic QA/QC summary forms (see Appendix I), and submitted to shift supervisor. The tabular printouts with strip charts and calibration data are filed together by analyte in the Lachat raw data file located in the Classical Chemistry offices.

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- 14.2 Case narratives should include information received on corrective action reports, any holding time problems, and any other problems associated with the analysis of samples.
- 14.3 The reporting limit or PQL for ammonia is 0.04 mg/L. Two significant figures are used for results less than 1 mg/L and three for results greater than or equal to 1.00 mg/L.
- 14.4 Deliverables and data review:
- 14.4.1 Level 1 Deliverable - Level 1 consists of tabular results as printed off of LIMS, holding times are verified for each sample. The chain of custody is checked for completeness. A case narrative and approval report is required for every job. The wet chemistry QA/QC summaries are not required but should be included if available.
 - 14.4.2 Level 2 or NJ II Deliverable - A level 2 package consists of tabular results, an approval report, laboratory chronicles, a case narrative, all QA/QC summaries (including method blank results) and a review of the raw data. The laboratory chain-of-custody is checked for completeness.
 - 14.4.3 CLP or NJ Tier I Deliverable - A CLP deliverable consists of a level 2 package plus a copy of the raw data.
 - 14.4.4 NYSDEC 91 Deliverables - A NYSDEC package consists of a CLP package plus NYSDEC '87 sample preparation and analysis forms. NYSDEC '91 deliverable forms include: cover page, and forms, I, II, III, IV, IVA, V, VI, and VII.

NOTE: Lab chronicles are not required for NYSDEC packages.

15.0 SUPPLEMENTAL DOCUMENTS

- 15.1 Methods Manual for the QuikChem Automated Ion Analyzer, Lachat Instruments, 1990.
- 15.2 Operating Manual for the QuikChem Automated Ion Analyzer, Lachat Instruments, 1990.
- 15.3 Analysis of Ammonia-Nitrogen in Environmental Samples, WC:021690.

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16.0 REFERENCES

- 16.1 Methods of Chemical Analysis of Water and Wastes, EPA 600, method 350.1, 1983.

17.0 SUBSTANTIVE REVISIONS

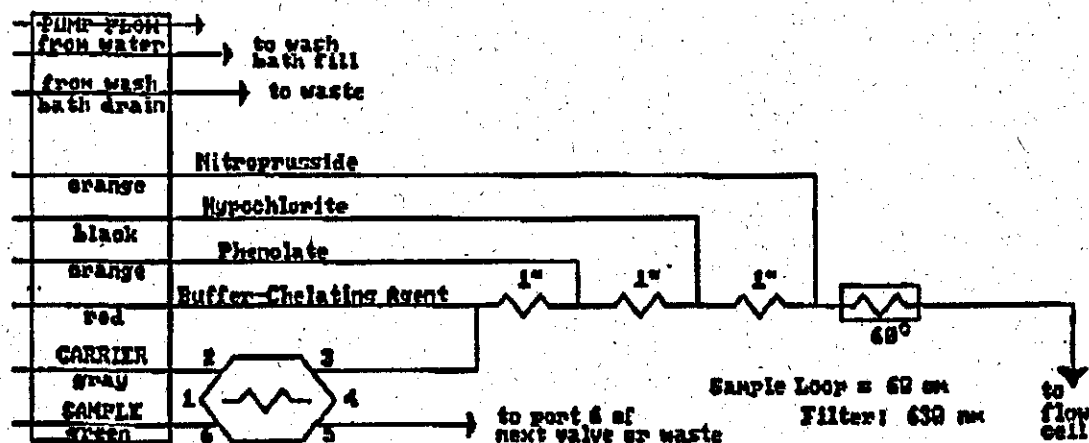
- 17.1 This SOP is an original document.

WC:070792:0

Appendix I

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Figure 1: Ammonia System Schematic



CARRIER is helium degassed water.

1"	is	70.0	cm of tubing on a 1 in coil support
2"	is	135	cm of tubing on a 2 in coil support
2.5"	is	168	cm of tubing on a 2.5 in coil support
3"	is	202	cm of tubing on a 3 in coil support
4"	is	255	cm of tubing on a 4 in coil support
8"	is	550	cm of tubing on a 8 in coil support

Heated tubing is shown inside a box with the temperature next to the box. Heated tubing is 650 cm unless otherwise specified.

All manifold tubing is 0.8 mm (0.032 in) i.d. This is 5.2 uL/cm.

IEA
200 Monroe Turnpike
Monroe, CT 06468 (203) 452-8200
CLASSICAL CHEMISTRY QA/QC SUMMARY

Analyte: _____ Date: _____ Ref: _____

LABORATORY CONTROL SAMPLE

Reference	True Value	Value Found	% Recovery

SAMPLE SPIKE

Lab Sample ID	Spike Result	Sample Result	Amount Added	% Recovery

DUPLICATES

Lab Sample ID	Result 1	Result 2	Mean	RPD

PREP BLANK

Blank ID	Result

Samples in Batch:	Comments:

AR300158

**IEA**

An Aquarion Company

200 Monroe Turnpike
Monroe, Connecticut 06468Phone 203-261-4458
Fax 203-268-5346**CORRECTIVE ACTION REPORT**QA:11108616
Rev. 06/02/92

Client : _____

Date : _____

Job/Case : _____

Reported by: _____

Sample(s) : _____

Group : _____

Category of Problem:

☐ Preservative
☐ Sample Matrix
☐ Holding Time
☐ Communication
☐ Other _____☐ Protocol
☐ Missing Sample
☐ Re-extraction

Description of Problem / Recommended Corrective Action :

Group(s) To Provide Corrective Action : _____
Reply To Originator Required ? : _____

Corrective Action Taken :

Distribution: _____

Florida
205-846-1730Schaumburg,
Illinois
708-705-0740N. Billerica,
Massachusetts
617-272-8212Whippany,
New Jersey
201-428-8181Research Triangle Park,
North Carolina
919-677-0090Essex Junction,
Vermont
802-878-9135

AR300159

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Figure 2: Ammonia Development Order.

<u>Cup #</u>	<u>Analytical ID</u>
1	ICV
2	ICB
3	LCS
4	CCV
5	CCB
6	PB
7	
8	
9	
10	Check Std (CCV)
11	CCB
12	
13	
14	
15	
16	
17	
18	
19	
20	Check Std (CCV)
21	CCB

Cups 7-9, 12-19, 22-29, ect. are for client samples including sample matrix quality control. All QC criteria of section 10.0 must be met within the development run.

AR300160



IEA

An Aquarion Company

200 Monroe Turnpike
Monroe, Connecticut 06468

Phone 203-261-4458
Fax 203-268-5346

STANDARD OPERATING PROCEDURE

TOTAL ORGANIC CARBON

WC:021390:1

Florida
305-846-1730

Schaumburg,
Illinois
708-705-0740

N. Billerica,
Massachusetts
617-272-8212

Whippany,
New Jersey
201-428-6181

Research Triangle Park,
North Carolina
919-677-0090

Essex Junction,
Vermont
802-878-6138

AR300161

WC:021390:1

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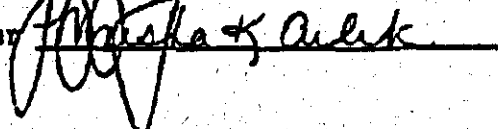
- 1.0 Approvals
- 2.0 Scope and Application
- 3.0 Summary of Method
- 4.0 Interferences
- 5.0 Safety
- 6.0 Sample Preservation and Storage
- 7.0 Apparatus and Materials
- 8.0 Reagents and Standard Preparation
- 9.0 Calibration
- 10.0 Quality Control
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- 12.0 Calculations
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- 16.0 References
- 17.0 Substantive Revisions
- Appendix I: Forms

AR300162

WC:021390:1

**STANDARD OPERATING PROCEDURE
TOTAL ORGANIC CARBON****1.0 APPROVALS**

The signature of the following individuals indicates that this SOP is complete and meets the requirements specified in corporate Document #QAS00200.NET.

Laboratory Director Quality Assurance Manager **2.0 SCOPE AND APPLICATION**

- 2.1 This method is applicable to determine the concentration of non-purgeable organic carbon in groundwater, surface and saline waters, and domestic and industrial wastes.
- 2.2 This method is applicable only to carbonaceous matter which is either soluble or has a particle size of 0.2 mm or less.
- 2.3 This method is based on EPA Method 415.2 and SW-846 Method 9060, and is suitable for the measurement of TOC above 0.50 mg/L.

3.0 SUMMARY OF METHOD

- 3.1 Organic carbon in a sample is converted to carbon dioxide utilizing a UV/persulfate catalytic oxidation technique. The carbon dioxide which is formed is measured by an infrared detector. The amount of carbon dioxide detected is directly proportional to the concentration of carbonaceous material in the sample.

4.0 INTERFERENCES

- 4.1 Inorganic carbon will interfere if it is not removed from the sample. The inorganic carbon can be removed by acidifying the sample with phosphoric acid and sparging out the CO₂ that was formed from the inorganic carbon.
- 4.2 This procedure is applicable only to homogeneous samples which can be injected into the apparatus reproducibly by means of a microli-ter-type syringe. The opening of the syringe limits the maximum size of particle which may be included in the sample.
- 4.3 Removal of inorganic carbon by acidification and purging can result in the loss of volatile organic substances.

WC:021390:1

5.0 SAFETY

- 5.1 At a minimum, wear PVC gloves, a lab coat and eye protection whenever handling samples, standards or reagents.
- 5.2 Always refer to your IEA Employee Safety Handbook prior to performing laboratory operations.

6.0 SAMPLE CONTAINERS AND PRESERVATION

- 6.1 Samples may be collected in either polyethylene or glass bottles (amber glass septa bottles are preferred). A minimum sample volume of 200 mL is to be collected for TOC analysis.
- 6.2 Water samples should be preserved with sulfuric acid (pH <2) and kept under refrigeration at 4°C until analysis.
- 6.3 All samples should be analyzed within 28 days of the date of collection, or 26 days from receipt for NYSDEC Protocols.

7.0 APPARATUS AND MATERIALS

- 7.1 Dohrmann DC-80 carbon analyzer
- 7.2 Culture tubes (18 x 150 mm)
- 7.3 Volumetric pipets
- 7.4 Volumetric flasks
- 7.5 Volumetric graduated syringes

8.0 REAGENTS AND STANDARD PREPARATION

- 8.1 All chemicals are to conform to the specifications set by the Reagent Chemical Committee of the American Chemical Society. Laboratory prepared "nano" water, which meets ASTM type II requirements, is used whenever reagent water is required.
- 8.2 Potassium hydrogen phthalate, stock solution, 2,000 mg/L carbon - Dissolve 0.425 g of potassium hydrogen phthalate (KHP) (primary standard grade) in nanopure water in a 100 mL volumetric flask. Add 0.2 mL concentrated phosphoric acid (H_3PO_4) and take to volume with nanopure water. Store in amber glass bottle and keep refrigerated. Make fresh monthly.

WC:021390:1

- 8.3 Potassium hydrogen phthalate, standard solution, 10 mg/L - Pipet 1.0 mL KHP stock solution into a 200 mL volumetric flask. Acidify with H_3PO_4 and fill to volume with nano H_2O . Make fresh daily.
- 8.4 Potassium hydrogen phthalate, standard solution, 20 mg/L - Pipet 1.0 mL KHP stock solution into a 100 mL volumetric flask. Acidify with H_3PO_4 and fill to volume with nano H_2O . Make fresh daily.
- 8.5 Potassium hydrogen phthalate, standard solution, 50 mg/L - Pipet 2.5 mL KHP stock solution into a 100 mL volumetric flask. Acidify with H_3PO_4 and fill to volume with nano H_2O . Make fresh daily.
- 8.6 Potassium hydrogen phthalate, standard solution, 1.0 mg/L - Pipet 10 mL of 10 mg/L standard solution into a 100 mL volumetric flask. Acidify with H_3PO_4 and fill to volume with nano H_2O . Make fresh daily.
- 8.7 Acidified Persulfate Reagent, 2% - Dissolve 20.0 g potassium persulfate ($\text{K}_2\text{S}_2\text{O}_8$) in 1 L of nanopure water. Add 1.0 mL H_3PO_4 and mix well. Store in amber glass.
- 8.8 Oxygen
- 8.9 Laboratory Control Standard (LCS) - Prepared following the independent source supplier's instructions.

9.0 CALIBRATION

- 9.1 The IR on the DC-80 should be on at all times. Turn all other power controls "on" and let it warm up for 30 minutes before use. Turn oxygen on and make sure regulator is set for 38 psi. Check persulfate in reactor vessel and put persulfate feed line in the potassium persulfate reagent bottle. Close pressure fingers down on pump tubing. Be sure that the two waste lines are draining into a waste bottle.
- 9.2 Wait for the baseline to stabilize to between 0.0100 and 0.0250 on the display. If the baseline does not stabilize within the appropriate range adjust the zero setting to compensate.
- 9.3 Before calibration check to make sure the calibration light is off. The DC-80 should be calibrated daily. The mode switch should be set for TOC. The sample volume switch should be set for 1 mL. The sample volume switch should be at 1 mL for direct injection of samples and for utilizing the autosampler tray.

WC:021390:1

- 9.4 Calibrate using the 10 mg/L standard. Refer to the Dohrmann DC-80 TOC Systems Manual for calibration information. Be sure baseline is stable and "ready" light is on before injecting standards. For direct injection of samples and standards, the 1,000 uL syringe is used. Spurge standards and samples for at least 2 minutes before injecting. The first three standards on the autosampler tray should also be sparged prior to set-up. The rest of the tray will be sparged as it goes along. Consult system manual for use of autosampler information.
- 9.5 Once the DC-80 is in calibration, a 5-point curve (including the blank) should be run (in duplicate) from the standards previously prepared. Nanopure water should be injected between samples and its value should be ≤ 0.50 mg/L. Samples should be run in quadruplicate. The recovery of each standard must be within 10 percent to accept the calibration.
- 9.6 Continuing Calibration
- 9.6.1 After every 10 samples at the end of the analytical run, a continuing calibration verification (CCV) is analyzed. The CCV is a 10 mg/L standard and must meet a ± 10 percent constraint in order to continue the analysis.
- 9.6.2 A continuing calibration blank (CCB) is analyzed following every CCV. The CCB is reagent water and must report at < 0.50 mg/L in order to continue the analysis.

10.0 QUALITY CONTROL

- 10.1 The reporting or practical quantitation limit (PQL) for total organic carbon in water is 0.50 mg/L. An MDL study performed on 02/01/91 yielded a 0.11 mg/L detection limit. The method performance for 1991 yielded an average relative percent difference of 2.15 percent, an average spike yield of 98.1 percent and an average LCS recovery of 103.8 percent.
- 10.2 The working range for the DC-80 TOC is 0.50 to 50 mg/L.
- 10.3 All stock solutions and standard preparations are logged and coded. All solutions are labeled with the following: analyte, concentration, analyst's initials, date prepared and expiration date.
- 10.4 All chemicals will conform to minimum specifications set by the Reagent Chemicals Committee of the American Chemical Society. All chemical inventories are used on a first-in/first-out basis.

WC:021390:1

10.5 Preparation Blank (PB)

10.5.1 A PB is reagent water that is treated as an analytical sample throughout the entire analytical process. A PB is analyzed with every batch of 10 or fewer samples.

10.5.2 If a PB's concentration exceeds the PQL, all samples that were prepared with the PB and have a concentration greater than the PQL and less than 10 times the concentration of the PB must be rerun.

10.6 Laboratory Control Sample (LCS)

10.6.1 One independent standard (LCS) is to be analyzed with each batch of 10 or fewer samples. The control limits for the LCS are ± 15 percent of the true value.

10.6.2 If the LCS recovery is outside the control limits, all samples associated with the LCS must be reanalyzed.

10.7 Duplicates

10.7.1 One duplicate analysis should be analyzed with every batch of 10 or fewer samples. Samples identified as field blanks may not be used for duplicate analysis. The relative percent difference (RPD) is calculated in Section 12.2. A control limit of 20 percent for RPD shall be applied for sample and duplicate values equal to or greater than 5 times the PQL.

10.8 Matrix Spikes

10.8.1 A sample spike is performed on every batch of 10 or fewer samples. Samples identified as field blanks may not be used for sample spike analysis. The sample matrix spike is calculated in Section 12.3.

The acceptable spike recovery range is 75-125 percent, if the sample concentration is greater than 4 times the spiking level, no criteria is placed on the spike recovery.

10.9 If both the spike and duplicate fail, all associated samples must be reanalyzed. If only the spike or the duplicate fail, a matrix interference is suspected and the failed QC is reanalyzed to confirm the results.

10.10 The batch size listed for the PB, LCS, duplicate and matrix spike, along with samples run in quadruplicate are for SW-846 protocols. For EPA 600 or SOP work the batch size is expanded to 20 samples.

WC:021390:1

- 10.11 A corrective action report (CAR) must be filed if any problems are associated with the analysis or any deviation of this SOP occurs (See Appendix I).

11.0 SAMPLE PREPARATION AND PROCEDURE

- 11.1 All samples to be analyzed must be signed out of sample control on the internal chain of custody (COC) for each job. All TOC data collected is recorded in the TOC log (See Appendix I).
- 11.2 See Section 9.0 for instrument set-up and daily calibration.
- 11.3 Analysis Procedure
- 11.3.1 Check the pH of an aliquot of the sample with pH paper. If the sample is not acidified, add phosphoric or concentrated sulfuric acid to lower the pH below 2.
- 11.3.2 Sparge the sample for a period of at least 3 minutes.
- 11.3.3 After the baseline has returned, the instrument will beep and the ready light will be on. At this time 1 mL of the sample should be injected into the injection septa. As soon as the injection is completed, the start button should be pushed.
- 11.3.4 Record the TOC measurement at the end of each injection run.
- 11.4 Samples are run either in duplicate or in quadruplicate depending on the analytical request. Nano blanks are run between samples to ensure that no carryover is taking place. TOC readouts of greater than 50.0 mg/L require dilution.
- 11.5 Never inject sample if the "ready" light is not on. If the error light comes on, an error message will be printed on the printout tape. The most common error is a timeout error. This means the sample peak is still tailing after the full 8 minutes it had to resolve. The sample should be diluted and rerun. Refer to system manual for error descriptions and troubleshooting information.
- 11.6 Instrument shutdown includes turning off the system power control, removing pressure from the pump fingers, turning off the oxygen, and removing and elevating the persulfate feed line.

WC:021390:1

11.7 Preventive Maintenance

11.7.1 The pump tubes, internal and external plumbing, septa, copper/tin scrubber and reaction vessel are checked daily and replaced as required.

11.7.2 The IR cell is cleaned periodically as required.

11.7.3 The spare parts inventory includes (at a minimum) pump tubes, flow restrictors, septa, syringes and perm driers.

12.0 CALCULATIONS

12.1 The TOC readout reports the TOC concentration directly in mg/L as carbon. The only data manipulation required is if the sample was diluted;

$$(\text{TOC readout}) \times (\text{dilution factor}) = \text{sample concentration} \\ (\text{mg/L as C})$$

12.2 The relative percent difference is calculated:

$$\text{RPD} = \frac{(S - D)}{(S + D)/2} \times 100$$

where: S = original sample result
D = duplicate sample result

12.3 The sample matrix spike is calculated:

$$\text{Percent recovery} = \frac{(\text{SSR} - \text{SR}) \times 100}{\text{SA}}$$

where: SSR = spiked sample result (mg/L)
SR = sample result (mg/L)
SA = spike added (mg/L)

13.0 ACCEPTANCE OF DATA

13.1 The technical data review includes verifying that samples analyzed were unpreserved and that holding times were met. All quality control measures are checked to ensure that all QC criteria described in Section 10.0 were met. The raw data is examined for completeness including a secondary signoff. Results entered into LIMS are checked for transcription errors against the raw data.

WC:021390:1

14.0 REPORTING OF RESULTS

- 14.1 Batch quality control is reported on the inorganic QA/QC summary forms (See Appendix I).
- 14.2 Case narratives should include information received on corrective action reports, any holding time problems, and any other problems associated with the analysis of samples.
- 14.3 The reporting limit or PQL for TOC is 0.50 mg/L. Two significant figures are used for results less than 1.00 mg/L, three are used for results greater than or equal to 1.00 mg/L.
- 14.4 Deliverables and data review:
 - 14.4.1 Level 1 Deliverable - Level 1 consists of tabular results as printed off of LIMS, holding times are verified for each sample. The chain of custody is checked for completeness. A case narrative and approval report is required for every job. The wet chemistry QA/QC summaries are not required, but should be included if available.
 - 14.4.2 Level 2 (NJ II Deliverable) - A level 2 package consists of tabular results, an approval report, laboratory chronicles, a case narrative, all QA/QC summaries (including method blank results) and a review of the raw data. The laboratory chain of custody is checked for completeness.
 - 14.4.3 CLP (NJ Tier I Deliverable) - A CLP deliverable consists of a level 2 package plus a copy of the raw data.
 - 14.4.4 NYSDEC '91 Deliverables - A NYSDEC package consists of a CLP package plus NYSDEC '87 sample preparation and analysis forms. NYSDEC '91 deliverable forms include: cover page and forms I, II, III, IV, IVA, V, VI and VII.

Note: Lab chronicles are not required for NYSDEC packages.

15.0 SUPPLEMENTAL DOCUMENTS

- 15.1 DC-80 TOC manual, Dohrmann, 13th Edition, 1989.

16.0 REFERENCES

- 16.1 Methods for Chemical Analysis of Water and Wastes, EPA 600, Method 415.2, 1983.
- 16.2 Test Methods for Evaluating Solid Waste, SW-846, 3rd Edition, Method 9060, 1986.

WC:021390:1

16.3 Standard Methods for the Examination of Water and Wastewater, 15th Edition, Method 505B, 1980.

17.0 SUBSTANTIVE REVISIONS

17.1 The format of this SOP was changed on 07/06/92 to correspond to document #QAS00200.NET.

APPENDIX I

AR300172

IEA
200 Monroe Turnpike
Monroe, CT 06468 (203) 452-8200

CLASSICAL CHEMISTRY QA/QC SUMMARY

Analyte: _____ Date: _____ Ref: _____

LABORATORY CONTROL SAMPLE

Reference	True Value	Value Found	% Recovery

SAMPLE SPIKE

Lab Sample ID	Spike Result	Sample Result	Amount Added	% Recovery

DUPLICATES

Lab Sample ID	Result 1	Result 2	Mean	RPD

PREP BLANK

Blank ID	Result

Samples in Batch:	Comments:

AR300174

**IEA**

An Aquarion Company

200 Monroe Turnpike
Monroe, Connecticut 06468Phone 203-261-4458
Fax 203-268-5346**CORRECTIVE ACTION REPORT**QA11108616
Rev. 06/02/92

Client : _____

Date : _____

Job/Case : _____

Reported by: _____

Sample(s) : _____

Group : _____

Category of Problem: _____

____ Preservative
____ Sample Matrix
____ Holding Time
____ Communication
____ Other _________ Protocol
____ Missing Sample
____ Re-extractionDescription of Problem / Recommended Corrective Action :

_____Group(s) To Provide Corrective Action : _____
Reply To Originator Required ? : _____Corrective Action Taken :

Distribution: _____

Asa,
Florida
305-846-1730Schaumburg,
Illinois
708-705-0740N. Billerica,
Massachusetts
617-272-6212Whippany,
New Jersey
201-428-8181Research Triangle Park,
North Carolina
919-677-0090Essex Junction,
Vermont
802-878-6138

AR300175



an environmental testing company

200 Monroe Turnpike
Monroe, Connecticut 06468
(203) 261-4458
FAX (203) 268-5348

2nd group
to Shuone - O
49 pages

STANDARD OPERATING PROCEDURE

ANALYSIS OF TOTAL ALKALINITY

IN WATER SAMPLES

WC:040491:0

Whippany,
New Jersey
201-428-8181

Schaumburg,
Illinois
708-705-8748

Miramar,
Florida
407-888-0928

Essex Junction,
Vermont
802-878-5138

N. Billerica,
Massachusetts
617-275

AR300176

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- 12.0 References

Appendix I: Forms

AR300177

Total Alkalinity

1.0 SCOPE AND APPLICATION

- 1.1 This method is applicable to drinking, surface, and saline waters, domestic and industrial wastes.
- 1.2 The method has a practical quantitation limit (PQL) of 1.0 mg/L (as CaCO_3) and is suitable for all higher concentration ranges of alkalinity. However, aliquots should be used to avoid a titration volume of greater than 50mL.
- 1.3 Automated titrametric analysis is equivalent.

2.0 SUMMARY OF METHOD

- 2.1 A sample is titrated with a standardized acid to an electrometrically determined end point of pH 4.5. The sample must not be filtered, or altered in any way.

3.0 INTERFERENCES

- 3.1 Organic material, soaps, oily matter, suspended soils, or precipitates may coat the glass electrode and cause a sluggish response.
- 3.2 Allow sufficient time between titrant additions to let the electrode response to come to equilibrium.

4.0 APPARATUS AND MATERIALS

- 4.1 A pH meter or electrically operated titrator that uses a glass electrode and can be read to 0.05 pH units. Standardize and calibrate according to manufacturer's instructions. If automatic temperature compensation is not provided, perform total alkalinity analysis at $25 \pm 2^\circ\text{C}$.
- 4.2 Titration rack and electrode support
- 4.3 Magnetic stirrer plate and bar magnets.
- 4.4 Pipets (Class A); 3, 5, 10, and 20mL.
- 4.5 Erlenmeyer flasks (wide mouth); 100 and 250mL.

WC:040491:0

AR300178

- 4.6 Volumetric flasks; 200 and 1000mL.
- 4.7 Burets (Class A); 50, 25, and 10mL.
- 4.8 Graduated cylinders; 25 and 100mL.

5.0 REAGENTS

NOTE: See 10.2 for acid normality calculation.

- 5.1 Sodium carbonate solution (approx. 0.05N):
Place 2.5 ± 0.2 grams (to the nearest mg) Na_2CO_3 (dried at 250°C for 4 hours and cooled in desiccator) into a 1 liter volumetric flask and brought to volume.

- 5.2 Standard sulfuric acid (0.1 N):
Dilute 3.0 mL of concentrated H_2SO_4 to 1 liter with reagent grade water. Standardize versus 40.0 mL of 0.05 N Na_2CO_3 solution with 60 mL of reagent grade water by titrating potentiometrically to a pH of approx. 5. Lift the electrode and rinse with reagent grade water into the beaker. Boil the solution gently for 3 to 5 minutes under a watch glass. Cool to room temperature. Rinse the watch glass into the beaker and continue the titration to the inflection point.

- 5.3 Standard sulfuric acid (0.02 N):
Dilute 200.0 mL of 0.1000 N H_2SO_4 to 1 liter with reagent grade water. Standardize by the potentiometric titration of 15.0 mL 0.05 N Na_2CO_3 solution as in 5.2.

CAUTION: Sulfuric acid solutions are corrosive and cause burns. Avoid all contact. Wear gloves and appropriate eye protection at all times!

6.0 SAMPLE STORAGE AND HANDLING

- 6.1 Samples are to be stored unpreserved at 4°C .
- 6.2 Samples are to be analyzed within 14 days of sampling or within 12 days of receipt (NYSDEC protocol).

7.0 PROCEDURE

- 7.1 All samples to be analyzed must be signed out of sample control on the chain-of-custody (COC) for each job. All alkalinity data collected is recorded in the alkalinity log (see appendix I).

WC:040491:0

AR300179

- 7.2 Place 100 mL or an appropriate smaller sample volume into a flask, measure the pH of the sample. Record the sample volume used.

NOTE: Use a sufficiently large sample size to require a volume of titrant (>10 mL in 50 mL buret) which will obtain good precision while keeping the volume low enough to permit a sharp end point.

- 7.3 Add 0.0200 N H_2SO_4 , being careful to stir thoroughly but gently to allow for equilibrium. Titrate to pH 4.5 and record the volume of titrant and exact normality.

NOTE: For samples containing >1000 mg/L as CaCO_3 of alkalinity, use 0.1000 N H_2SO_4 as the titrant.

8.0 QUALITY CONTROL

- 8.1 All stock solutions and standard preparations are logged and coded. All solutions are labeled with the following: analyte, concentration, analyst's initials, date prepared, and expiration date.

- 8.2 All chemicals will conform to minimum specifications set by the Reagent Chemicals Committee of the American Chemical Society. All chemical inventories are used on a first-in, first-out basis.

- 8.3 A prep blank (reagent water) is to be analyzed with every batch of ten or fewer samples.

- 8.4 One laboratory control sample (LCS) is to be analyzed with each batch of ten or fewer samples. LCS control limits are $\pm 15\%$ of the known concentration.

NOTE: Should the LCS fail, the source of the problem must be corrected and an acceptable LCS run before continuing with the analysis of samples.

- 8.5 One sample duplicate must be analyzed from each batch of ten or fewer samples. Samples identified as field blanks are not to be used for duplicate analysis. The relative percent difference (RPD) between the sample and duplicate must be less than or equal to 20.0 (see section 10.4 for the RPD calculation). No RPD criteria is applied if both results are less than five times the PQL. No RPD criteria is applied if one result is lower than five times the PQL and one above, if the difference between the two results is no more than the PQL.

WC:040491:0

AR300180

9.0 METHOD PERFORMANCE

- 9.1 The average relative percent difference over the last 25 positive duplicates was 2.36% as of 3/28/91.
- 9.2 The last ten LCS percent recoveries averaged 105.6% as of 3/28/91.

10.0 DATA HANDLING AND REDUCTION

- 10.1 All raw data including sample volume, titrant volume, and acid normality are entered into LIMS. The LIMS system calculates the results.
- 10.2 The normality of the H_2SO_4 titrant is calculated:

$$N = \frac{A \times B}{53. \times C}$$

where: A = grams Na_2CO_3 into 1 liter
B = mL Na_2CO_3 solution
C = mL acid used to inflection point

- 10.3 Manual alkalinity calculation (titration to pH 4.5).

$$\text{Alkalinity (mg/L as } CaCO_3) = \frac{A \times N \times 50,000}{\text{mL of sample}}$$

where: A = mL of acid titrated
N = normality of acid

- 10.4 The relative percent difference is calculated:

$$RPD = \frac{(S - D)}{(S + D)/2} \times 100$$

where: S = Original sample result
D = Duplicate sample result

- 10.5 The sample quality control and LCS recovery results are reported on the QA/QC summary form (see appendix I).
- 10.6 The technical data review includes verifying that samples analyzed were unpreserved and that holding times were met. All quality control measures are checked to insure that all QC criteria were met. Results entered into LIMS are checked for transcription error against the raw data.

WC:040491:0

AR300181

11.0 DOCUMENTATION OF PROBLEMS

- 11.1 Corrective Action Reports - must be filed if any problems are encountered during analysis.
- 11.2 Case Narratives - should include information collected from corrective action reports and any deviations from this SOP.

12.0 REFERENCES

- 12.1 Methods of Chemical Analysis of Water and Wastes, EPA 600, Method 310.1, 1983.
- 12.2 STANDARD METHODS: For the Examination of Water and Wastewater, 15th ed., Method 403, 1980.

WC:040491:0

AR300182

03/14/95

15:14

203 268 5346

IEA CONNECTICUT

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APPENDIX I

AR300183

INORGANICS QA/QC SUMMARY

Analyte: _____ Date: _____

Notebook Ref: _____

DUPLICATES

Lab Sample ID	Result 1	Result 2	Mean	RPD

BLANKS

Blank ID	Result

Samples in Batch:	Comments:

AR300184



an environmental testing company

300 Monroe Turnpike
Monroe, Connecticut 06468
(203) 281-4458
FAX (203) 288-5346

CORRECTIVE ACTION REPORT

QA:111086:5
Rev. 06/20/91

Client : _____

Date : _____

Job/Case : _____

Analyst: _____

Sample(s): _____

Group : _____

Category of Problem:

☐ Preservative
☐ Sample Matrix
☐ Holding Time
☐ Communication
☐ Other _____

☐ Protocol
☐ Missing Sample
☐ Re-extraction

Description of Problem / Recommended Corrective Action :

Group(s) To Provide Corrective Action : _____

Reply To Originator Required ? : _____

Corrective Action Taken :

Miami,
Florida
1-800-8928

Schaumburg,
Illinois
708-705-8740

N. Billerica,
Massachusetts
617-372-9217

Whippany,
New Jersey
201-428-8181

Cary,
North Carolina
919-677-0000

East Jordan,
Vermont
802-678-9136

AR300185



an environmental testing company

200 Monroe Turnpike
Monroe, Connecticut 06468
(203) 261-4458
FAX (203) 268-5346

STANDARD OPERATING PROCEDURE

ANALYSIS OF BIOCHEMICAL OXYGEN DEMAND

IN WATER

WC:110790:1

Whippany,
New Jersey
201-426-8181

Schaumburg,
Illinois
708-705-8740

Stromer,
Florida
407-988-0828

Essex Junction,
Vermont
802-876-5138

N. Andover,
Massachusetts
617-872-8212

Cary,
North Carolina
919-877-0000

AR300186

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WC:110790:1

AR300187

Biochemical Oxygen Demand (405.1)

1.0 SCOPE AND APPLICATION

- 1.1 The biochemical oxygen demand (BOD) test is used for determining the relative oxygen requirements of waste municipal and industrial waters. Application of the test to organic waste discharges allows calculation of the effect of the discharges on the oxygen resources of the receiving water.
- 1.2 BOD determines the oxygen required for the degradation of organics and the oxygen used to oxidize inorganic material. Unless a nitrogen inhibitor is used BOD may also measure the oxygen required to oxidize reduced forms of nitrogen.

2.0 SUMMARY OF METHOD

- 2.1 The sample of waste, or an appropriate dilution, is incubated for five days at 20 C in the dark. The reduction in dissolved oxygen concentration during the incubation period yields a measure of the biochemical oxygen demand.
- 2.2 Only the five day BOD is described in this procedure, variations of biochemical oxygen demand including shorter or longer incubation periods may be requested.

3.0 INTERFERENCES

- 3.1 The BOD dilution water is buffered to ensure a pH suitable for bacterial growth, if this condition is not met proper oxygen reduction will not occur.
- 3.2 Samples containing toxic substances may kill or slow the seed bacteria growth, such samples may require special study and treatment.

4.0 APPARATUS AND MATERIALS

- 4.1 Buret, 50 mL
- 4.2 Pipets, Class A

WC:110790:1

AR300188

- 4.3 Incubation bottles, 300 mL
- 4.4 Incubators (temp. 20 C)
- 4.5 Pipets, disposable
- 4.6 Dissolved Oxygen Probe and Meter
- 4.7 Airators
- 4.8 Erlenmeyer Flasks, 500 mL

5.0 REAGENTS

- 5.1 Phosphate buffer solution: Dissolve 8.5 g KH_2PO_4 , 21.75 g K_2HPO_4 , 33.4 g $\text{Na}_2\text{HPO}_4 \cdot 7\text{H}_2\text{O}$, and 1.7 g NH_4Cl in approx. 500 mL of reagent grade water and dilute to 1 liter.
- 5.2 Magnesium sulfate solution: In approx. 500 ml of reagent grade water dissolve 22.5 g $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$ and dilute to 1 liter.
- 5.3 Calcium chloride solution: Dissolve 27.5 g CaCl_2 in reagent grade water and dilute to 1 liter.
- 5.4 Ferric chloride solution: Dissolve 0.25 g $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$ in reagent grade water and dilute to 1 liter.
- 5.5 Nitrification inhibitor: 2-chloro-6-(trichloromethyl) pyridine (Nach 2533).
- 5.6 Glucose-glutamic acid solution: Dry reagent grade glucose and glutamic acid at 103 C for 1 hour. Add 0.150 g glucose and 0.150 g glutamic acid to reagent grade water and dilute to 1 liter. Prepare fresh weekly.
- 5.7 Seed: Commercially available Polyseed.
- 5.8 Manganous sulfate solution: Dissolve 400 g $\text{MnSO}_4 \cdot 2\text{H}_2\text{O}$ in reagent grade water and dilute to 1 liter.
- 5.9 Alkali-azide reagent: Dissolve 10 g NaN_3 in 500 mL of reagent grade water. Add 480 g NaOH and 750 g NaI, and stir until dissolved.
- 5.10 Sulfuric acid, Conc.
- 5.11 Starch solution: Dissolve 2 g reagent grade starch and 0.2 g salicylic acid (as a preservative) in 100 mL hot distilled water.

WC:110790:1

AR300189

5.12 Spiking solution: Dissolve 0.425 mg of Potassium hydrogen phthalate in reagent grade water and dilute to 1 liter.

5.13 Standard Sodium Thiosulfate, 0.0250 N

6.0 SAMPLE PRESERVATION AND STORAGE

6.1 Samples collected for biochemical oxygen demand are to be stored unpreserved at 4 C. The holding time for BOD is 48 hours from the time of sampling or 24 hours from the time of receipt for NYSDEC 89 protocols.

7.0 PROCEDURE

7.1 Preparation of dilution water: To the required volume of dilution water add 1 mL of phosphate buffer, Ferric chloride, Calcium chloride, and Magnesium sulfate solutions per liter of reagent grade water. If only the measurement of carbonaceous BOD is desired, add 10 mg of nitrification inhibitor per liter of dilution water. The water is to be aerated for 1 hour using a small air pump. The dilution water is to be prepared daily.

7.2 Seed preparation: Dissolve 1 polyseed capsul in 500 mL of prepared dilution water (Prepare Daily).

7.3 Standardize the dissolved oxygen meter: Collect two BOD bottles of water, use one to titrate and the other to calibrate the meter once the dissolved oxygen is known (in mg/L).

Add 2.0 mL of Manganous sulfate solution, Alkali-azide reagent, and conc. H_2SO_4 . Stopper carefully to leave no head space, mix well.

Titrate 201 mL of the prepared solution with 0.0250 N sodium thiosulfate to a pale yellow color. Add a few drops of starch and continue titrating to the first disappearance of blue.

NOTE: When titrating with 0.0250 N titrant:
1 mL titrant = 1 mL Dissolved Oxygen.

Calibrate the D.O. meter following the manufactures instructions and the known dissolved oxygen from the titration above.

WC:110790:1

AR300190

- 7.4 Prepare the following control bottles: blank dilution water, 20 mL seed, 5 mL glucose solution, 5 mL glucose solution and 2 mL seed, 10 mL glucose solution, 10 mL glucose solution and 2 mL seed. Dilute to volume with dilution water.
- 7.5 Prepare samples at several dilutions, ranging by an order of magnitude where possible. Seed all of the sample bottles and dilute to volume with dilution water. Prepare dilutions that will provide a residual D.O. of 1.0 mg/L and a D.O. depletion of at least 1.0 mg/L (2 mg/L is desirable).

NOTE: The samples should be removed from the coolers to allow them to warm to room temperature.

The experienced analyst can often determine the proper dilutions to run based on historical data and the physical nature of the sample. List below are examples of dilutions their corresponding BOD values, based on no seed correction.

Table 1. Dilution Factors compared to BOD Results

Sample Volume (mL)	Dil. Factor	D.O. Depletion (mg/L)	BOD (mg/L)
0.1	3000	2.0	6000
0.5	600	2.0	1200
1.0	300	2.0	300
5.0	60	2.0	120
10.	30	2.0	60
20.	15	2.0	30
25.	12	2.0	24
50.	6	2.0	12
100	3	2.0	6
150	2	2.0	4
150	2	1.0	2
200	1.5	2.0	3
300	1	2.0	2

- 7.6 Read the initial dissolved oxygen using the calibrated meter and stopper the bottles immediately after the D.O. has been read.

After all initial dissolved oxygen measurements have been taken, place the bottles in the BOD incubator for five days.

WC:110790:1

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7.7 After five days, recalibrate the D.O. meter and record the final dissolved oxygen.

8.0 QUALITY CONTROL

8.1 All stock standard preparations are logged and coded. All standards are labeled with the following: date, initials, analyte, and concentration.

8.2 All chemicals should conform to specifications set by the Reagent Chemicals Committee of the American Chemical Society. All chemical inventories are used on a first in first out basis.

8.3 A sample prep. blank (dilution water) is analyzed with each batch of twenty or fewer samples. The dissolved oxygen depletion should not exceed 0.2 ppm.

8.4 Laboratory Control Sample (LCS)

Glucose-glutamic acid check serves as the LCS and is to be analyzed with each batch of twenty or fewer samples. The acceptable glucose-glutamic acid check range is 200 ± 37 mg/L, if this standard is out of criteria seek corrective action.

8.5 One sample duplicate must be analyzed from each batch of twenty or fewer samples. Samples identified as field blanks are not to be used for duplicate analysis. The relative percent difference (RPD) between the sample and duplicate must be less than or equal to 20.0 (see section 10.4 for the RPD calculation). No RPD criteria is applied if both results are less than five times the PQL. No RPD criteria is applied if one result is lower than five times the PQL and one above, if the difference between the two results is no more than the PQL.

8.6 A sample spike is performed on every batch of twenty or fewer samples (see 10.5 for spike recovery calc.) Samples identified as field blanks may not be used for sample spike analysis. The acceptable spike recovery range is 75 to 125%, if the sample concentration is greater than 4 times the spiking level, no criteria is placed on the spike.

WC:110790:1

AR300192

9.0 METHOD PERFORMANCE

- 9.1 The RPD over the last 50 duplicates was 6.9% for waters as of 4/1/91.
- 9.2 The average spike recovery over the same period of time was 93.6% for waters.
- 9.3 No data available for any other matrix.

10.0 DATA HANDLING AND REDUCTION

- 10.1 All raw data including sample volume, seed correction, initial and final D.O. concentrations are entered into the LIMS system. The LIMS system calculates the results.
- 10.2 The reporting or practical quantitation limit (PQL) for waters is 2.0 mg/L.
- 10.3 Manual Calculation:

$$\text{BOD mg/L} = \frac{(D_1 - D_2) - (B_1 - B_2)f}{P}$$

where: D_1 = initial sample DO (mg/L)
 D_2 = final sample DO (mg/L)
 B_1 = initial seeded blank DO (mg/L)
 B_2 = final seeded blank DO (mg/L)
 P = decimal volumetric fraction of sample used
 f = ratio of seed in sample to seed in control

- 10.4 The relative percent difference is calculated:

$$\text{RPD} = \frac{(S - D)}{(S + D)/2} \times 100$$

where: S = Original sample result
 D = Duplicate sample result

- 10.5 The sample matrix spike is calculated:

$$\text{Percent Recovery} = \frac{(\text{SSR} - \text{SR}) \times 100}{\text{SA}}$$

where: SSR = Spiked Sample Result (mg/L)
 SR = Sample Result (mg/L)
 SA = Spike Added (mg/L)

WC:110790:1

AR300193

- 10.6 The sample quality control and prep blank results are reported on the QA/QC summary form (see appendix I).
- 10.7 The technical data review includes verifying that samples analyzed were unpreserved and that holding times were met. All quality control measures are checked to insure that all QC criteria were met. Results entered into LIMS are checked for transcription error against the raw data.
- 11.0 DOCUMENTATION OF PROBLEMS
- 11.1 Corrective Action Reports - must be filed if any problems are associated with the analysis (see appendix I).
- 11.2 Case Narratives - should include any deviation from this SOP or any difficulties encountered in the procedure.
- 12.0 REFERENCES
- 12.1 Methods of Chemical Analysis of Water and Wastes, EPA 600, Method 405.1, 1983.
- 12.2 STANDARD METHODS: For the Examination of Water and Wastewater, 15th edition, Method 507, 1980.

WC:110790:1

4R300194

03/14/95

15:21

203 288 5348

1EA CONNECTICUT

0020

APPENDIX I

AR300195

IEA
200 Monroe Turnpike
Monroe, CT 06468 (203) 452-8200

CLASSICAL CHEMISTRY QA/QC SUMMARY

Analyte: _____

Date: _____

Ref: _____

LABORATORY CONTROL SAMPLE

Reference	True Value	Value Found	% Recovery

SAMPLE SPIKE

Lab Sample ID	spike Result	Sample Result	Amount Added	% Recovery

DUPLICATES

Lab Sample ID	Result 1	Result 2	Mean	RPD

PREP BLANK

Blank ID	Result

Samples in Batch:	Comments:

AR300196

IEA/CT

Group(s): _____

Date: _____

CORRECTIVE ACTION REPORT

Problem/Question reported by: _____

Job/Case Number: _____ Sample Number(s): _____

Description of Problem: _____

_____Summary of Corrective Action(s): _____

Is this a recurring Problem? _____

Should SOP be modified or updated? _____

Cooperative help of the following groups/people is requested: _____

Approval of Group/Section leader: _____

Distribution: Section/Group Leader
Lab Manager
QA Officer
Other: _____

AR300197

**IEA**

An Aquarion Company

200 Monroe Turnpike
Monroe, Connecticut 06468Phone 203-261-4458
Fax 203-268-5348

STANDARD OPERATING PROCEDURE

ANALYSIS OF HARDNESS IN WATER

WC:081390:1Sunrise,
Florida
305-846-1730Schaumburg,
Illinois
708-705-0740N. Billerica,
Massachusetts
617-272-5212Whippany,
New Jersey
201-426-8181Research Triangle Park,
North Carolina
919-677-0090Essex Junction,
Vermont
802-878-5138

AR300198

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AR300199

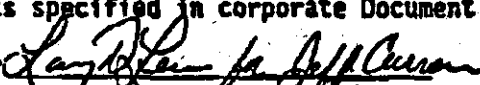
WC:081390:1

**STANDARD OPERATING PROCEDURE
ANALYSIS OF HARDNESS IN WATER**

1.0 APPROVALS

The signature of the following individuals indicates that this SOP is complete and meets the requirements specified in corporate Document #QAS00200.NET.

Laboratory Director



Quality Assurance Manager

**2.0 SCOPE AND APPLICATION**

- 2.1 This method is used as a measure of hardness in a sample and is applicable to all waters and wastewaters. The method is based on EPA Method 130.2.
- 2.2 The method is suitable for all concentration ranges of hardness, however in order to avoid large titration volumes, use a sample aliquot containing not more than 25 mg CaCO_3 .

3.0 SUMMARY OF METHOD

- 3.1 Ethylenediaminetetraacetic acid and its sodium salts (EDTA) form a chelated soluble complex when added to a solution of certain metal cations. If a small amount of dye such as Eriochrome Black T or Calmagite is added to an aqueous solution containing calcium and magnesium ions at a pH of 10.0 ± 0.1 , the solution becomes wine red. If EDTA is added as a titrant, the calcium and magnesium will be complexed, and when all of the magnesium and calcium has been complexed the solution turns from wine red to blue, marking the end point of the titration. Magnesium ion must be present to yield a satisfactory end point. To ensure this, a small amount of complexometrically neutral magnesium salt of EDTA is added to the buffer; this automatically introduces sufficient magnesium and obviates the need for a blank correction.

The sharpness of the end point increases with increasing pH, however the pH cannot be increased indefinitely because of the danger of precipitating calcium carbonate, CaCO_3 , or magnesium hydroxide, $\text{Mg}(\text{OH})_2$, and because the dye changes color at high values. The specified pH of 10.0 ± 0.1 is a satisfactory compromise. A limit of 5 minutes is set for the duration of the titration to minimize the tendency toward CaCO_3 precipitation.

WC:081390:1

4.0 INTERFERENCES

- 4.1 Some metal ions interfere by causing fading or indistinct end points due to stoichiometric consumption of EDTA. Reduce this interference by adding certain inhibitors before titration. When higher concentrations of heavy metals are present, determine calcium and magnesium by a non-EDTA method and obtain hardness by calculation (Section 12.3).

5.0 SAFETY

- 5.1 At a minimum, wear PVC gloves, a lab coat and eye protection when handling samples and reagents.
- 5.2 Always refer to your IEA Employee Safety Handbook prior to performing lab operations.

6.0 SAMPLE CONTAINERS AND PRESERVATION

- 6.1 Samples may be collected in either polyethylene or glass bottles. A minimum sample volume of 200 mL are to be collected for hardness analysis.
- 6.2 Water samples should be acidified to a pH of less than 2 by the addition of concentrated nitric acid and kept under refrigeration at 4°C until they are analyzed.
- 6.3 All samples should be analyzed within 6 months of the date of collection.

7.0 APPARATUS AND MATERIALS

- 7.1 Buret, 50 mL
- 7.2 Erlenmeyer flasks
- 7.3 Stirring bars
- 7.4 Stir plates
- 7.5 Pipets, Class A
- 7.6 Graduated cylinders, 100 and 250 mL
- 7.7 Volumetric flasks, 1 L

WC:081390:1

8.0 REAGENTS AND STANDARD PREPARATION

- 8.1 All chemicals are to conform to specifications set by the Reagent Chemicals Committee of the American Chemical Society. Laboratory prepared "nano" water is used whenever reagent water is required.
- 8.2 Buffer solution - Dissolve 1.179 g of disodium EDTA (analytical grade) and 780 mg of $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$ (or 644 mg $\text{MgCl}_2 \cdot 6\text{H}_2\text{O}$) in 50 mL of DI water. Add this solution to a 250 mL volumetric flask containing 16.9 g of ammonium chloride (NH_4Cl) and 143 mL concentrated ammonium hydroxide (NH_4OH) with mixing and dilute to the mark with DI water. Store in a plastic bottle for no longer than one month. An acceptable alternative is a commercially available stable buffer, such as the "Hach Hardness 1 Product", which has a 6-month shelf life.
- 8.3 Inhibitor solutions - These are to be used only if interferences are evident during the titration. Please check with lab supervisor or lab manager first.
- 8.3.1 Inhibitor I: NaCN powder
- Note: This is an extremely poisonous chemical, in the presence of acids HCN can be liberated. This indicator should only be used in the presence of a senior staff chemist.
- 8.3.2 Inhibitor II: Dissolve 5.0 g of $\text{Na}_2\text{S} \cdot 9\text{H}_2\text{O}$ in 100 mL of DI water. Cover with tightly fitted rubber stopper.
- 8.3.3 Inhibitor III: Dissolve 4.5 g of hydroxylamine hydrochloride in 100 mL of 95 percent ethanol or isopropanol.
- 8.4 Indicator solutions - A Calmagite indicator solution can be purchased commercially.
- 8.5 Standard EDTA titrant, 0.02 N - Place 3.723 g of analytical reagent grade disodium ethylenediaminetetraacetate dihydrate, $\text{Na}_2\text{H}_2\text{C}_{10}\text{H}_{12}\text{O}_8\text{N}_2 \cdot 2\text{H}_2\text{O}$ in a 1 liter volumetric flask and dilute to the mark with DI water. Check with standard calcium solution by titration. (See Section 1 under procedure.) Store in polyethylene.
- 8.6 Standard calcium solution - Place 1.000 g of anhydrous calcium carbonate in a 500 mL flask. Slowly add 1:1 HCl until all of the CaCO_3 has dissolved. Cool. Add a few drops of methyl red indicator and adjust to intermediate orange color by adding 3N ammonium hydroxide (NH_4OH) or 1:1 HCl as required. Quantitatively transfer to a 1 liter volumetric flask and dilute to the mark with DI water.

WC:081390:1

- 8.7 Hydrochloric acid solution, 1:1.
- 8.8 Methyl red indicator - Dissolve 0.10 g methyl red in DI water in a 100 mL volumetric flask and dilute to the mark.
- 8.9 Ammonium hydroxide solution, 3N - Dilute 210 mL of concentrated ammonium hydroxide (NH_4OH) to 1 liter with DI water.
- 8.10 Ammonium hydroxide solution, 1N - Dilute 70 mL of concentrated ammonium hydroxide to 1 liter with DI water.
- 8.11 Laboratory control standard - Prepare following independent source supplier's instructions.

9.0 CALIBRATION

Before starting on the samples, make sure that the EDTA solution has been standardized. To standardize the EDTA solution, follow the procedure outlined below.

- 9.1 Place 10.0 mL of standard calcium solution in a vessel containing about 50 mL of DI water. Add 1 mL of buffer solution and 1 to 2 drops of indicator. Titrate slowly with continuous stirring with the EDTA until the last reddish tinge disappears. Add the last few drops at 3 to 5 second intervals. At the endpoint the color is blue. Total titration duration should be 5 minutes from the time of the buffer addition. Perform this titration in duplicate. Calculate the normality of the EDTA as shown below.

$$N \text{ of EDTA} = (0.20)/(\text{mL of EDTA added})$$

10.0 QUALITY CONTROL

- 10.1 The practical quantitation limit (PQL) for hardness is 1.0 mg/L. A method detection limit (MDL) study conducted on 02/01/91 yielded a 0.22 mg/L MDL.
- 10.2 The method performance for 1991 yielded a 4.56 average RPD and an average spike recovery of 97.8 percent over the same period.
- 10.3 Preparation blank (PB)
 - 10.3.1 A PB is reagent water that is treated as an analytical sample throughout the entire analytical process. A PB is analyzed with every batch of 20 or fewer samples.
 - 10.3.2 If a preparation blank's concentration exceeds the PQL, all samples that were prepared with the PB and have a concen-

WC:081390:1

tration greater than the PQL and less than 10 times the concentration of the PB must be rerun.

10.4 Laboratory control sample (LCS)

10.4.1 One independent standard (LCS) is to be analyzed with each batch of 20 or fewer samples. The control limits for the LCS are ± 15 percent of the true value.

10.4.2 If the LCS recovery is outside the control limits, all samples associated with the LCS must be reanalyzed.

10.5 Duplicates

10.5.1 One duplicate analysis should be analyzed with every batch of 20 or fewer samples. Samples identified as field blanks may not be used for duplicate analysis. The relative percent difference (RPD) is calculated as follows:

$$RPD = \frac{(S - D)}{(S + D)/2} \times 100$$

where: S = original sample value (mg/L)
D = duplicate sample value (mg/L)

A control limit of 20 percent for RPD shall be applied for sample and duplicate values equal to or greater than 5 times the PQL.

10.6 Spikes

10.6.1 A sample spike is performed on every batch of 20 or fewer samples. Samples identified as field blanks may not be used for sample spike analysis. The sample matrix spike is calculated:

$$\text{Percent recovery} = \frac{(SSR - SR)}{SA} \times 100$$

where: SSR = spiked sample result (mg/L)
SR = sample result (mg/L)
SA = spike added (mg/L)

The acceptable spike recovery range is 75-125 percent, if the sample concentration is greater than 4 times the spiking level, no criteria is placed on the spike recovery.

10.7 If both the spike and duplicate fail, all associated samples must be reanalyzed. If only the spike or the duplicate fail, a matrix in-

WC:081390:1

interference is suspected and the failed QC is reanalyzed to confirm the results.

- 10.8 A corrective action report (CAR) must be filed if any problems are associated with the analysis or any deviation of this SOP occurs (See Appendix I).

11.0 SAMPLE PREPARATION AND PROCEDURE

- 11.1 For drinking, ground, surface and saline waters and dilutions thereof, no pretreatment steps are necessary. Highly polluted wastewaters may require metals acid digestion prior to analysis (see supervisor).

11.2 Titration of sample normal-to-high hardness:

- 11.2.1 Sample should require <15 mL EDTA titrant (Section 8.5) and titration should be completed within 5 minutes of buffer addition.
- 11.2.2 Place 10 mL sample in titration vessels, neutralize with 1N ammonium hydroxide (Section 8.10) and dilute to about 50 mL.
- 11.2.3 Add 1 to 2 mL buffer solution (Section 8.2).
- 11.2.4 If end point is not sharp (as determined by practice run) add inhibitor at this point (Section 11.4).
- 11.2.5 Add 1 to 2 drops indicator solution (Section 8.4).
- 11.2.6 Titrate slowly with continuous stirring with standard EDTA titrant until last reddish tint disappears. Solution is normally blue at end point.

11.3 Titration of sample low hardness (less than 5 mg/L):

- 11.3.1 Use a larger sample (50 mL)
- 11.3.2 Use a microburet and run a blank amount using nano reagent water.

11.4 To correct for interferences:

- 11.4.1 Some metal ions interfere by causing fading or indistinct end points. The use of inhibitors reduces these effects. The inhibitor selection is based on the metals present, consult with supervisor to determine which inhibitor to use.

WC:081390:1

12.0 CALCULATIONS

12.1 All raw data including sample volume, titrant volume and normality are entered into LIMS. The LIMS system calculates the results.

12.2 Manual hardness calculation:

$$\text{Hardness (EDTA) as mg CaCO}_3\text{/L} = \frac{A \times B \times 100}{\text{ml sample}}$$

where: A = mL titration for sample

B = mg CaCO₃ equivalent to 1.00 mL EDTA titration

12.3 If interferences require, hardness can be calculated from calcium and magnesium metals data.

$$\text{Hardness (mg/L as CaCO}_3\text{)} = 2.497 (\text{Ca, mg/L}) + 4.118 (\text{Mg, mg/L})$$

13.0 ACCEPTANCE OF DATA

13.1 The technical data review includes verifying that samples were preserved and holding times were met. All standard and batch quality control are checked to ensure that they were within criteria. All results entered into LIMS are checked for transcription errors against the raw data.

14.0 REPORTING OF RESULTS

14.1 Batch quality control is reported on the inorganic QA/QC summary forms (See Appendix I).

14.2 Case narratives should include information received on corrective action reports, any holding time problems, and any other problems associated with the analysis of samples.

14.3 The reporting limit or PQL for hardness is 1.0 mg/L. Two significant figures are used for results less than 10 mg/L, three for results greater than 10 mg/L.

14.4 Deliverables and data review:

14.4.1 Level 1 Deliverable - A level 1 consists of tabular results as printed off of LIMS, holding times are verified for each sample per analyte. The chain-of-custody is checked for completion. A case narrative and approval report is required for every job. The classical chemistry QA/QC summaries are not required but should be included if available.

WC:081390:1

Note: All subcontracted IEA level 1 jobs require lab chronicles!

14.4.2 Level 2 or NJ Tier II Deliverable - A NJ tier II consists of tabular results, an approval report, lab chronicles, a case narrative, all QA/QC summaries (including method blank results) and a review of the raw data. The lab chain of custody may be checked for completeness.

14.4.3 CLP or NJ Tier I Deliverable - A CLP or tier I deliverable consists of a level 2 package plus a copy of the raw data.

14.4.4 NYSDEC '91 Deliverables - NYSDEC '91 deliverables consist of a CLP package plus NYSDEC '87 sample preparation and analysis forms. NYSDEC '91 packages also include NYSDEC deliverables forms which include: cover page, and forms I, II, III, IV, IVA, V, VI and VII.

Note: Lab chronicles are not required for NYSDEC packages.

15.0 SUPPLEMENTAL DOCUMENTS - N/A

16.0 REFERENCES

16.1 Methods of Chemical Analysis of Water and Wastes, EPA 600, 1983, Method 130.2.

17.0 SUBSTANTIVE REVISIONS

17.1 The format of this SOP was changed on 06/21/92 to correspond to document #QAS00200.NET.

03/14/95

15:29

203 268 5346

1EA CONNECTICUT

4000

APPENDIX I

200 VICTORIE TULPINE - VICTORIE CONNECTICUT 05-468 - 12031-452 6200

AR300208

Hardness

IEA
200 Monroe Turnpike
Monroe, CT 06468 (203) 452-8200

Sample ID	Batch ID	Sample Volume	Buret Measurement		mls Titrated	Hardness mg/L as CaCO ₃
			Initial	Final		
	PB					
	LCS					
	DUP					
	SPIKE					
	1					
	2					
	3					
	4					
	5					
	6					
	7					
	8					
	9					
	10					
	11					
	12					
	13					
	14					
	15					
	16					
	17					
	18					
	19					
	20					

Stock Code ID _____
Spike Amount Added _____

Analyst _____
Date _____

Page _____

Reviewed by _____

AR300209

IEA
200 Monroe Turnpike
Monroe, CT 06468 (203) 452-8200
CLASSICAL CHEMISTRY QA/QC SUMMARY

Analyte: _____ Date: _____ Ref: _____

LABORATORY CONTROL SAMPLE

Reference	True Value	Value Found	% Recovery

SAMPLE SPIKE

Lab Sample ID	Spike Result	Sample Result	Amount Added	% Recovery

DUPLICATES

Lab Sample ID	Result 1	Result 2	Mean	RPD

PREP BLANK

Blank ID	Result

Samples in Batch:	Comments:

AR300210



an environmental testing company

200 Menree Turnpike
Menree, Connecticut 06468
(203) 261-4456
FAX (203) 268-5346

CORRECTIVE ACTION REPORT

QA:111056:3
Rev. 06/20/91

Client : _____

Date : _____

Job/Case : _____

Analyst: _____

Sample(s) : _____

Group : _____

Category of Problem:

☐ Preservative
☐ Sample Matrix
☐ Holding Time
☐ Communication
☐ Other _____

☐ Protocol
☐ Missing Sample
☐ Re-extraction

Description of Problem / Recommended Corrective Action :

Group(s) To Provide Corrective Action : _____

Reply To Originator Required ? : _____

Corrective Action Taken :

Middletown,
Florida
305-940-0928

Schaumburg,
Illinois
708-701-0740

N. Billerica,
Massachusetts
617-673-5213

Whippany,
New Jersey
908-438-6161

Greensboro,
North Carolina
919-677-0000

Green Junction,
Vermont
802-670-6136

AR300211

**IEA**

An Aquarion Company

200 Monroe Turnpike
Munroe, Connecticut 06468Phone 203-261-4458
Fax 203-268-5348

STANDARD OPERATING PROCEDURE

ANALYSIS OF SULFATE IN WATER

WC:030489:1Sunrise,
Florida
305-846-1730Schaumburg,
Illinois
708-705-0740N. Attleboro,
Massachusetts
617-272-3212Whippany,
New Jersey
201-428-8181Research Triangle Park,
North Carolina
919-677-0090Essex Junction,
Vermont
802-878-5138

AR300212

WC:030489:1

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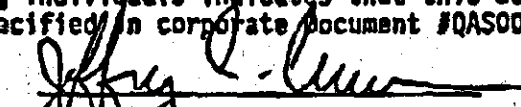
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- 2.0 Scope and Application
- 3.0 Summary of Method
- 4.0 Interferences
- 5.0 Safety
- 6.0 Sample Preservation and Storage
- 7.0 Apparatus and Materials
- 8.0 Reagents and Standard Preparation
- 9.0 Calibration
- 10.0 Quality Control
- 11.0 Sample Preparation and Procedure
- 12.0 Calculations
- 13.0 Acceptance of Data
- 14.0 Reporting of Results
- 15.0 Supplemental Documents
- 16.0 References
- 17.0 Substantive Revisions
- Appendix I: Forms

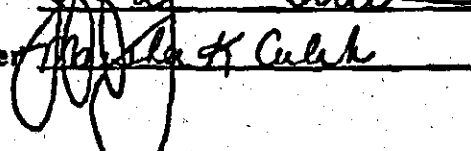
AR300213

WC:030489:1

**STANDARD OPERATING PROCEDURE
ANALYSIS OF SULFATE IN WATER****1.0 APPROVALS**

The signature of the following individuals indicates that this SOP is complete and meets the requirements specified in corporate document #QAS00200.NET.

Laboratory Director 

Quality Assurance Manager 

2.0 SCOPE AND APPLICATION

- 2.1 This method is applicable to determine the concentration of sulfate in drinking, surface and saline waters and domestic and industrial wastes.
- 2.2 This method is applicable to the measurement of sulfate above 10.0 mg/L, and is based on EPA Method 375.3 and Standard Methods 426B.

3.0 SUMMARY OF METHOD

- 3.1 Sulfate is precipitated as barium sulfate in a hydrochloric acid medium by the addition of barium chloride. After a period of digestion, the precipitate is filtered, washed with warm water, dried and weighed as BaSO_4 .

4.0 INTERFERENCES

- 4.1 High results may be obtained for samples containing nitrate, sulfite and silica. Samples containing suspended matter are filtered prior to analysis. Low results may be obtained from alkali sulfates, due to occlusion with barium sulfate. Alkali sulfates decompose when heated. Heavy metals such as chromium and iron may interfere by causing incomplete precipitation and precipitation of heavy metal sulfates.

5.0 SAFETY

- 5.1 At a minimum, wear PVC gloves, a lab coat and eye protection whenever handling samples, standards or reagents.
- 5.2 Always refer to your IEA Employee Safety Handbook prior to performing laboratory operations.

WC:030489:1

6.0 SAMPLE PRESERVATION AND STORAGE

- 6.1 A minimum sample volume of 200 mL must be collected in either polyethylene or glass bottles for sulfate analysis.
- 6.2 Samples are to be collected unpreserved and stored at 4°C until the time of analysis.
- 6.3 All samples must be analyzed within 28 days of the date of collection. The NYSDEC '91 holding time is 26 days from the verified time of receipt.

7.0 APPARATUS AND MATERIALS

- 7.1 Gooch crucibles
- 7.2 Analytical balance (± 0.1 mg)
- 7.3 Drying oven (with thermostatic controls)
- 7.4 Desiccator
- 7.5 Filters, 934AH
- 7.6 Filters, membrane (0.45 μ m)
- 7.7 Hot plates
- 7.8 Graduated cylinders, 100 mL
- 7.9 Pipets, class A (5 and 10 mL)
- 7.10 Filtering apparatus
- 7.11 Crucibles (previously ignited at 800°C)
- 7.12 Filter paper, ashless (#42)
- 7.13 Muffle furnace

8.0 REAGENTS AND STANDARD PREPARATION

- 8.1 Methyl Red Indicator Solution - Dissolve 100 mg of methyl red sodium salt in 100 mL DI water, or 100 mg of methyl red and 2-3 drops of 2N sodium hydroxide.
- 8.2 Hydrochloric Acid 1+1 - Add 250 mL of concentrated HCl to 250 mL DI water. CAUTION: PERFORM UNDER HOOD. HCL VAPORS!

WC:030489:1

- 8.3 Barium Chloride Solution - Dissolve 100 grams of $\text{BaCl}_2 \cdot 2\text{H}_2\text{O}$ in 1 liter of DI water. Filter through a 0.45μ filter paper. One ml of this reagent is capable of precipitating approximately 40 mg SO_4 .
- 8.4 Standard Sulfate (1,000 ppm) - Weigh 1.479 g of dried sodium sulfate (105°C) and dissolve in 1 liter of DI water (1 ml = 1 mg SO_4). This solution is stable for 3 months.

9.0 CALIBRATION

- 9.1 Each time an analytical balance is used a 20 g Class S weight is measured.
- 9.2 Any problems with an analytical balance must be reported to the area supervisor immediately.

10.0 QUALITY CONTROL

- 10.1 The reporting or practical quantitation limit (PQL) for sulfate is 10.0 mg/L. An MDL study performed on 03/14/90 yielded a 6.70 mg/L detection limit.
- 10.2 The working range for this sulfate procedure is 10.0 to 500 mg/L without dilution.
- 10.3 All stock solutions and standard preparations are logged and coded. All solutions are labeled with the following: analyte, concentration, analyst's initials, date prepared and expiration date.
- 10.4 All chemicals will conform to minimum specifications set by the Reagent Chemicals Committee of the American Chemical Society. All chemical inventories are used on a first-in/first-out basis.
- 10.5 Preparation Blank (PB)
- 10.5.1 A PB is reagent water that is treated as an analytical sample throughout the entire analytical process. A PB is analyzed with every batch of 20 or fewer samples.
- 10.5.2 If a PB's concentration exceeds the PQL, all samples that were prepared with the PB and have a concentration greater than the PQL and less than 10 times the concentration of the PB must be rerun.

WC:030489:1

10.6 Laboratory Control Sample (LCS)

10.6.1 One independent standard (LCS) is to be analyzed with each batch of 20 or fewer samples. The control limits for the LCS are ± 15 percent of the true value.

10.6.2 If the LCS recovery is outside the control limits, all samples associated with the LCS must be reanalyzed.

10.7 Duplicates

10.7.1 One duplicate analysis should be analyzed with every batch of 20 or fewer samples. Samples identified as field blanks may not be used for duplicate analysis. The relative percent difference (RPD) is calculated in Section 12.2. A control limit of 20 percent for RPD shall be applied for sample and duplicate values equal to or greater than 5 times the PQL.

10.8 Matrix Spikes

10.8.1 A sample spike is performed on every batch of 20 or fewer samples. Samples identified as field blanks may not be used for sample spike analysis. The sample matrix spike is calculated in Section 12.3.

The acceptable spike recovery range is 75-125 percent, if the sample concentration is greater than 4 times the spiking level, no criteria is placed on the spike recovery.

10.9 If both the spike and duplicate fail, all associated samples must be reanalyzed. If only the spike or the duplicate fail, a matrix interference is suspected and the failed QC is reanalyzed to confirm the results.

10.10 Batch size and control limit requirements may be superseded or modified by a client's quality assurance plan for a specific project, check with supervisor about specific projects.

10.11 A corrective action report (CAR) must be filed if any problems are associated with the analysis or any deviation of this SOP occurs (See Appendix I).

11.0 SAMPLE PREPARATION AND PROCEDURE

Two procedures are outlined here for sulfate analysis. The first method is by drying and the second by ignition (after the precipitation of barium sulfate step). Analysis by strict 375.3 methodology is done by ignition.

AR300217

WC:030489:1

11.1 Precipitation Procedure

- 11.1.1 Filter a 100 mL aliquot of the sample through a 0.45 μ m membrane filter to remove suspended particulates.
- 11.1.2 Add 2 or 3 drops of methyl red indicator.
- 11.1.3 Add 1+1 HCl until the sample solution becomes red (pH 4.5-5).
- 11.1.4 Add 1-2 mL more of 1+1 HCl.
- 11.1.5 Heat the sample solutions to near boiling, while warm and with stirring add warm BaCl_2 solution.
- 11.1.6 If the amount of precipitate is small, add a total of 5 mL BaCl_2 solution.
- 11.1.7 Digest the sample solution at 80-90°C for at least 2 hours.

NOTE: A white precipitate denotes the presence of sulfate.

11.2 Analysis by Drying of Residue

- 11.2.1 Prepare Gooch crucibles by washing, fitting with two 934 AH filter pads and filtering through a half crucible of nano water to make sure the pads are snug. Place in drying oven for at least one hour, desiccate for one hour, and weigh on analytical balance. (Be sure the Gooch ID number is recorded.)
- 11.2.2 Filter the precipitate through a weighed Gooch crucible. Add another 5 mL BaCl_2 to the filtrate to ensure all the SO_4^{2-} has been precipitated out. If the filtrate turns white, filter it through the crucible again. Put the crucibles in the drying oven (at 105°C) for at least two hours, desiccate for at least two hours and weigh back on the same analytical balance.

11.3 Analysis by Ignition of Residue

- 11.3.1 Filter the precipitate solution through a 0.45 μ filter paper, using a vacuum filter apparatus. For samples with heavy precipitates Whatman 42 ashless filter paper pulp may be combined with the precipitate to aid filtration.
- 11.3.2 Rinse the sample container with warm DI water and transfer rinsate to vacuum filter.

WC:030489:1

- 11.3.3 Rinse the precipitate with three 10 mL portions of warm DI water to remove residual chloride.
- 11.3.4 Transfer the precipitate and filter paper to a weighed crucible that has been treated at 800°C. Record crucible weight on worksheet.
- 11.3.5 Dry the filter paper and precipitate.
- 11.3.6 Ignite the sample residue at 800°C for 1 hour. The filter paper should not flame.
- 11.3.7 Allow the residue to cool in a desiccator and weigh. Crucibles should be approximately 60°C before removing from furnace. Record final weight on worksheet.

12.0 CALCULATIONS

- 12.1 All raw data including sample volume, initial and final Gooch weights are entered into LIMS. The LIMS system calculates the results.

- 12.2 The relative percent difference is calculated:

$$RPD = \frac{(S - D)}{(S + D)/2} \times 100$$

where: S = original sample result
D = duplicate sample result

- 12.3 The sample matrix spike is calculated:

$$\text{Percent recovery} = \frac{(SSR - SR)}{SA} \times 100$$

where: SSR = spiked sample result (mg/L)
SR = sample result (mg/L)
SA = spike added (mg/L)

- 12.4 Manual Calculation of Sulfates in Water

$$SO_4 \text{ mg/L} = \frac{F - I}{V} \times 411.5$$

where: F = final weight of crucible + residue (in mg)
I = initial weight of crucible (in mg)
V = sample volume (in mL)

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13.0 ACCEPTANCE OF DATA

- 13.1 The technical data review includes verifying that samples analyzed were unpreserved and that holding times were met. All quality control measures are checked to ensure that all QC criteria described in Section 10.0 were met. The raw data is examined for completeness including a secondary signoff. Results entered into LIMS are checked for transcription errors against the raw data.

14.0 REPORTING OF RESULTS

- 14.1 Batch quality control is reported on the inorganic QA/QC summary forms (See Appendix I).
- 14.2 Case narratives should include information received on corrective action reports, any holding time problems, and any other problems associated with the analysis of samples.
- 14.3 The reporting limit or PQL for sulfate is 10.0 mg/L. Three significant figures are reported for all results.
- 14.4 Deliverables and data review:
- 14.4.1 Level 1 Deliverable - Level 1 consists of tabular results as printed off of LIMS, holding times are verified for each sample. The chain of custody is checked for completeness. A case narrative and approval report is required for every job. The wet chemistry QA/QC summaries are not required, but should be included if available.
- 14.4.2 Level 2 (NJ II Deliverable) - A level 2 package consists of tabular results, an approval report, laboratory chronicles, a case narrative, all QA/QC summaries (including method blank results) and a review of the raw data. The laboratory chain of custody is checked for completeness.
- 14.4.3 CLP (NJ Tier I Deliverable) - A CLP deliverable consists of a level 2 package plus a copy of the raw data.
- 14.4.4 NYSDEC '91 Deliverables - A NYSDEC package consists of a CLP package plus NYSDEC '87 sample preparation and analysis forms. NYSDEC '91 deliverable forms include: cover page and forms I, II, III, IV, IVA, V, VI and VII.

Note: Lab chronicles are not required for NYSDEC packages.

15.0 SUPPLEMENTAL DOCUMENTS - N/A

WC:030489:1

16.0 REFERENCES

- 16.1 Methods for Chemical Analysis of Water and Wastes, EPA 600, Method 375.3, 1983.
- 16.2 Standard Methods for the Examination of Water and Wastewater, 16th Edition, Methods 426A and 426B, 1985.

17.0 SUBSTANTIVE REVISIONS

- 17.1 The format of this SOP was changed on 07/08/92 to correspond to document #QAS00200.NET.

03/14/85

15:37

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APPENDIX I

AR300222

**IEA**

An Aquarion Company

200 Monroe Turnpike
Monroe, Connecticut 06468Phone 203-261-4458
Fax 203-268-5348**CORRECTIVE ACTION REPORT**QA:111086:6
Rev. 06/02/92

Client : _____ Date : _____
Job/Case : _____ Reported by: _____
Sample(s): _____ Group : _____

Category of Problem:

☐ Preservative	☐ Protocol
☐ Sample Matrix	☐ Missing Sample
☐ Holding Time	☐ Re-extraction
☐ Communication	
☐ Other _____	

Description of Problem / Recommended Corrective Action :

Group(s) To Provide Corrective Action : _____
Reply To Originator Required ? : _____

Corrective Action Taken :

Distribution: _____Sunrise,
Florida
305-846-1730Schaumburg,
Illinois
708-706-0740Rt. Bitterton,
Massachusetts
617-272-5212Whippany,
New Jersey
201-428-8191Research Triangle Park,
North Carolina
919-677-0030Essex Junction,
Vermont
802-878-5138

AR300224

***Attachment 2
Geotechnical Laboratory Quality
Assurance Plan***

AR300225

**Comprehensive Quality
Assurance Plan**

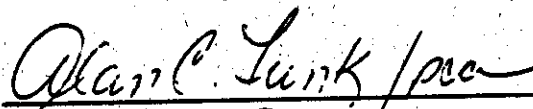
July 29, 1992

**Environmental Resources Management, Inc.
855 Springdale Drive
Exton, Pennsylvania 19341**

AR300226

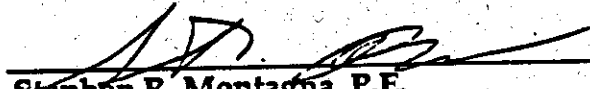
Comprehensive Quality Assurance Plan

July 29, 1992



Alan C. Funk, P.G.

**Department Manager, Geoenvironmental
Science and Engineering**



Stephen R. Montagna, P.E.

Technical Director

**Environmental Resources Management, Inc.
855 Springdale Drive
Exton, Pennsylvania 19341**

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Table 3-1 Calibration Table

Instrument	Initial Calibration		Continuing Calibration Verification	
	Frequency	Acceptance Criteria	Frequency	Acceptance Criteria
Sieve Analysis	At start of each test series	NBS calibrated glass beads used to calibrate sieves. See Section 7.2.5.	Every 20 tests	Percent retained must meet minimum ASTM criteria. Max variability $\leq$ 2%
Atterburg Limits	At start of each week	Cup drop must meet ASTM criteria. Max 1 CM $\pm$ 1mm	Every 5 tests	Must meet ASTM criteria. Max 1 cm $\pm$ 1mm
Hydrometer Analysis	At start of each week continuing calibration	Reading of 0 in column of distilled water $\pm$ 1 mm	Every 10 tests	Reading of 0 in columns of distilled water $\pm$ 1 mm
Unconfined Compression Analysis (UCC, CBR, etc.)	Each new run	Vertical speed calibration verification with $\pm$ 5% and lead cells calibrated within 5%	Every 5 tests	Same as Initial
Triaxial Compression Analysis (UU, CU, CD)	Each new run	Same as unconfined compression with chamber pressure cell as an added item. Chamber pressure cell and regulator calibrated within 5%	Every 5 tests	Same as Initial
Permeability Analysis		Same as Triaxial pressure cell calibration	Every 5 tests	Same as Initial
Consolidation Analysis		Incremental load readings within $\pm$ 5%	Every 5 tests	Same as Initial
Compaction Analysis		Hammer drop height within 5%.	Every 5 tests	Same as Initial
Balance	At start of each test series	Within 0.01 grams for analytical balance. Within 1.0 grams for top load balance	Every 5 tests	Same as Initial
pH Meter	Each new run	Utilize various buffer solutions. Variability within $\pm$ 0.1	Every test	Same as Initial

Organic Content Test and Bulk Density Test utilize balance equipment only.

Corrective Action

Check out of spec sieve for damage. Rerun calibration. If still out of spec, replace sieve

Reset cup drop. Check set screw for wear. Replace if worn. Check base for wear (denting). If dent exceeds 1 mm in depth, replace base

Check hydrometer for cracks or damage. Rerun calibration

Check dial gauge gearing for wear. If worn, replace. Check load cell for deformation and damage. If damaged or deformed, replace

Same as unconfined compression analysis. Check chamber pressure cell and regulator for damage or wear. If damaged or worn, replace. Check chamber for leaks

Same as Triaxial chamber cell check.

Check load arm hinge for wear and load weights for damage. If wear or damage is noted, replace

Check hammer for denting or elongation. If hammer drop criteria can't be met, replace.

Reset adjustment screen for proper reading on calibration weights

Try new buffer solution. Recheck, if criteria still can't be met, resubmit meter to manufacturer for repair

SAMPLE PROCEDURES

ERM, Inc. provides sampling services and sampling kits through a separate service not part of the geotechnical laboratory. However, some elements of sampling kits are part of the QA work within the geotechnical laboratory.

4.1

SAMPLING KITS

The technical director will determine the type and number of jars/containers needed for sampling, when provided with the number of sampling sites and the parameters to be analyzed. The jar/container will identify the client, site number, parameters, and space to indicate date, time, location, and any other information pertinent to the acquisition of the sample.

All jars/containers used for the collection of samples will be precleaned to EPA specifications by the supplier. Sampling kits will include the coolers needed to transport the samples when applicable. The client will receive a list of required jars/containers for the appropriate parameters (Figure 4-1). If the client requests, a chain-of-custody form (Figure 4-2) will be provided.

The field sampler is responsible for packaging the samples and adding ice to ensure that the samples remain cold during transportation. He/she must also complete, sign, and date the chain-of-custody form. The samples are in the custody of the sampler until they are relinquished to a common carrier or until the sampler transports the coolers to the laboratory. If the sampler sends the samples by a common carrier, this transfer of custody must be recorded on the chain-of-custody and the sampler should place a custody seal on each cooler before the carrier assumes possession.

5.0

SAMPLE CUSTODY

In order to demonstrate reliability of data which may be used as evidence in a legal case or required by a regulatory agency, an accurate written record tracing the possession of the sample from its receipt at the laboratory to the time of its disposal must be maintained. ERM, Inc. considers that samples to be in custody if any of the following states exists:

- In actual physical possession;
- In view after being in physical possession;
- In physical possession and locked up so no one can tamper with the sample;
- In a secured area, restricted to authorized personnel;
- Samples will be maintained in custody until the disposal.

5.1

LABORATORY SAMPLE RECEIPT

As the samples arrive at the laboratory, the laboratory manager will receive them. If the samples were picked up by our Warehouse Department, the warehouse supervisor must sign the chain-of-custody when receiving and relinquishing the samples. If a carrier service has been used, the receipt of shipment will be signed and the waybill will be retained for our files. At this time, a sample receipt checklist is initiated (Figure 5.1) for each shipment. This form is begun by the laboratory to initially record a shipment so that no samples will every be overlooked in case the shipment cannot be given immediate attention. The custody seal will remain intact until the laboratory manager is ready to log-in that particular set of samples.

Before the cooler is opened, the custody seal is inspected to ensure that the samples were not tampered with before the coolers' arrival at the laboratory. The sample receipt checklist is then completed for each shipment. The internal temperature of each cooler is taken and recorded prior to the samples' removal. The samples are checked against the chain-of-custody, as well as against the information provided by the ERM Project Manager in charge of that client's work.

Should any discrepancies occur among the expected incoming samples, the chain-of-custody form, and the received samples, the samples in question will be held by the laboratory manager without being logged in until the discrepancies

are resolved. The Project Manager for that client will be notified and will attempt to contact the client immediately. If the problem cannot be resolved in a minimum of time, the samples will be placed in a secure area which conforms to the required method of storage. The samples will not be logged in until the problems have been resolved.

After any inconsistencies have been reconciled, the client's instructions will be recorded on the Sample Receipt Checklist. The samples are now ready to be logged in.

5.2

SAMPLE LOG-IN

The laboratory manager will log the samples as soon as possible after receipt. Samples will be logged in within 24 hours after receipt with the following exceptions:

- Samples received during a holiday or between 5 pm on Friday and 8 am on Monday. Unless prior notification of incoming rush samples have been received, these samples will be logged in on the next normal work day.
- Samples submitted by clients without any indication of the tests to be performed or with unclear or incomplete information. Every effort will be made by the Project Manager to contact the client on the same day as sample receipt. If same day entry is not possible, any special storage requirements will be observed.

Each shipment of samples will be logged in as a new group unless specifically instructed to add a shipment of samples to an already existing group (e.g., ongoing sampling of one site). The group number is assigned consecutively whenever a new set of samples is logged in. Sample numbers are also assigned consecutively and consist of a unique identification number for each sample.

The form that is used to record all of the samples received and logged in: the Numerical Sample Log-In (Figure 5.2) is included which is a numerical listing of all samples logged in.

The information tracked by the login system includes the date of receipt in the laboratory, the date and time of sample collection, the initials of the sampler (if provided by the client), the laboratory identification of the sample, the requested analyses, and the date that results were promised to the client. Records also include the identity of the person who received the samples, the identity of the

person who logged in the samples and the identity of the project manager who approved the login.

5.2.1 Sample Storage and Security/Internal Custody

All samples are assigned a storage location when each group is logged in. This location is assigned based upon the required storage demands of each parameter. Each sample will remain in its storage location until the laboratory is ready to use it. When the laboratory manager removes a sample from storage, he/she must sign for it. Documentation is kept for each storage area (Figure 5.3, and 5.4). Sample handling is kept to a minimum. Technicians in possession of samples will remove the amount required and return the sample to storage with a minimum of delay. These forms will be stored by the laboratory manager to document sample security.

Should transfer of a sample to another laboratory be necessary, the laboratory manager will generate a chain of custody identifying the sample with the ERM laboratory identification and listing the required analyses and methods along with any special instructions. The sample will be placed in a cooler, will be cooled with ice if required, and transported by courier or common carrier as is appropriate.

5.3 RECORDKEEPING

All documents generated or received during any sample group's receipt or storage will be kept by the laboratory manager. Those forms which only record information relating to a single group of samples are filed in a folder created for that particular group of samples. These forms include the Chain-of-Custody, the Sample Receipt form, the Sample Receipt Checklist, and shipping documents. Any additional information supplied by the client or the Project Manager which concerns that particular group will also be retained in the group file. Those forms which record information relating to more than one group will be maintained in the laboratory manager's files (i.e., Sample Check Out Form, Numerical Sample Log-In) for a period of not less than six months. After that time the records will be archived under the direction of the technical director. The technical director is responsible for maintaining the security and inventory of the files which have been archived.

5.3.1 *Sample Tracking*

The laboratory manager is responsible for generating work assignment forms after the samples are logged. A copy of the Group Backlog Report which shows all tests assigned for a group, the identification and the storage location for all samples within the group, and the result due date, is sent to the technical director. Worksheets used by the laboratory technicians for reporting their results are also generated upon login and distributed by the laboratory manager.

The data of completion of each step of the analysis is tracked. The technicians who perform sample preparation steps such as drying or saturation must turn in the worksheets for the preparation steps. A Backlog Report by project is generated each week by the laboratory manager and distributed to all project managers and the technical director so work assignments can be tracked.

5.4 *SAMPLE DISPOSAL*

Once the final data report has been issued, any remaining sample will be kept in its assigned storage location for a period of at least 15 days. Any client who needs longer retention time must inform the project manager of these requirements. Normally, samples will be returned to the client or sampling location for disposal. If a client prefers that the laboratory dispose of the samples, the laboratory will contract a waste disposal company to dispose of those samples that have been determined to be hazardous. Project managers are given a form to complete (Figure 5.9) to serve as a record of the final disposition of the remaining samples. The method and date of disposal are filed in the group file which is maintained with the laboratory manager.

Section: 5
Date: 26 June 1992

Revision No.: 0

Figure 5-1 ERM, Inc. Sample Receipt Checklist

CLIENT: _____

CONTACT: _____

PROJECT: _____

TELEPHONE: _____

DATE SHIPPED: _____

CARRIER: _____

DATE RECEIVED: _____

RECEIVED BY: _____

NUMBER OF KITS RECEIVED: _____

TIME RECEIVED: _____

KIT CHECKLIST

KIT ID	COC PRESENT	CUSTODY TAPE PRESENT?	INTACT?	COOLER TEMP	# OF SAMPLE CONTAINERS

INCONSISTENCIES

SAMPLE	PARAMETER	INCONSISTENCY

ACTION TAKEN

PERSON CONTACTED: _____ DATE: _____

RESOLUTION: _____

ERM EMPLOYEE: _____ DATE: _____

SA152.01

The ERM Group

GAP 6/18/92

AR300234

Revision No.: 0

Numerical Sample Log-In

[illegible]

AR 300235

Figure 5-3 Sample Check Out Room Temp Storage

[illegible]

AR 300236

Figure 5-9 Sample Disposal Form

TO: ERM PROJECT MANAGER
FROM: GEOTECHNICAL LABORATORY
SUBJECT: SAMPLE DISPOSAL OF GROUP _____

THIS NOTICE IS TO INFORM YOU THAT GROUP _____ ID DUE FOR DISPOSAL ON _____

PLEASE CONTACT YOUR CLIENT REGARDING THIS MATTER, AS A TIMELY RESPONSE IS REQUESTED WITHIN THREE DAYS.

NO RESPONSE BY YOU WILL BE TREATED AS A HOLD ORDER, WHICH WILL ACCUMULATE STORAGE CHARGES FOR THE CLIENT.

ALSO, IT IS ENCOURAGED TO RETURN SAMPLES TO THE CLIENT WHENEVER POSSIBLE.

TO BE FILLED IN BY THE PROJECT MANAGER AND THE WHOLE FORM RETURNED TO THE GEOTECHNICAL LABORATORY.

CLIENT REPRESENTATIVE _____
CLIENT CONTACT DATE _____

SAMPLE SHOULD BE: _____ HELD PENDING FURTHER NOTICE.*
_____ DISPOSED OF IMMEDIATELY.**
_____ RETURN TO CLIENT. (INDICATE ADDRESS)

*THIS WILL ACCUMULATE A STORAGE CHARGE OF _____ PER _____.

**IF THE SAMPLE IS DEEMED TO BE HAZARDOUS, BASED ON OUR TESTING OR KNOWLEDGE OF THE SAMPLE, THERE WILL BE A DISPOSAL FEE ATTACHED TO THIS PROJECT.

Project Manager Approval _____

Date _____

SA155.00

AR300238

6.0

LABORATORY OPERATIONS

Glassware, Bottles, Pans and Mixing Bowls

The basic cleaning steps are:

- Removal of surface residuals immediately after use - As soon as possible after container (i.e., glassware, pans, bowls or bottles) has come in contact with sample, the container should be placed in the hot detergent soak.
- Hot soak to loosen and flotate particulate material - The hot soak consists of a bath of a suitable detergent in water of 50° C or higher. The detergent, powder or liquid, should be entirely synthetic and not a fatty acid base. There are very few areas of the country where the water hardness is sufficiently low to avoid the formation of some hard-water scum resulting from the reaction between calcium and magnesium salts with a fatty acid soap. This hard-water scum or curd would have an affinity particularly for many chlorinated compounds and, being almost wholly water-insoluble, would deposit on all glassware in the bath in a thin film.

There are many suitable detergents on the wholesale and retail market. Most of the common liquid dishwashing detergents sold at retail are satisfactory but are more expensive than other comparable products sold industrially. Alconox, in powder or tablet form, is manufactured by Alconox, Inc., New York, and is marketed by a number of laboratory supply firms. Sparkleen, another powdered product, is distributed by Fisher Scientific Company.

- Hot-water rinse to flush away flotated particulates.

6.1

WASTE DISPOSAL PROCEDURES

All wastes generated by the laboratory will be disposed of following all appropriate federal, state, and local laws. Wastes will be disposed of through a reputable and licensed hazardous waste disposal firm. Hazardous waste manifests will be maintained for at least thirty years after disposal.

Table 6-1 Recent Storage

Chemical
Mineral Acids

Method of Storage
Stored in original containers in
vented cabinet. Cabinet is in
air conditioned area of
laboratory.

Alcohol

Stored in original containers in
vented cabinet designed for
flammable chemicals. Cabinet is
in air conditioned area of
laboratory.

General Chemicals

Stored in original containers in
cabinet in laboratory. Cabinets
are segregated by lab storage
code. Cabinets are in air
conditioned area of laboratory.

7.0 CALIBRATION PROCEDURES AND FREQUENCY

7.1 INSTRUMENTATION/EQUIPMENT LISTS

The equipment used for performing analyses is listed in Table 7.1.

7.2 INSTRUMENT CALIBRATION

Individual instrument calibration will be addressed for the specific parameter(s) for which it is used. See Table 7-2 for expanded calibration information.

7.2.1 pH Meter

Calibration

The pH meter is calibrated prior to each use. Three standard buffer solutions are used. A pH 4.0 buffer is used first to standardize the meter, followed by a pH 10.0 buffer. A pH 7.0 buffer is used as a final check of the meter.

Standards

Scientific Products pH 4.0 Reference Buffer
Scientific Products pH 7.0 Reference Buffer
Scientific Products pH 10.0 Reference Buffer

7.2.2 Analytical Balances

Calibration

Each balance is checked before each use to ensure the accuracy of the instrument. Measurements are recorded in a bound notebook which is kept with the balance.

Standards

A set of Class S weights are used.

7.2.3 Toploading Balances

Calibration

The balances are checked before each use and at a frequency of 20 samples to ensure the accuracy of the instrument. Measurements are recorded in a bound notebook which is kept with each balance.

Standards

A set of Class P weights are used. All Class P weight sets are calibrated on an annual basis against a set of certified Class S weights.

7.2.4 Thermometers

Calibration

A correction factor is calculated for each thermometer and is attached to the appropriate temperature chart or attached directly to the thermometer, depending upon the use of the thermometer.

Standard

All thermometers are calibrated against an NBS-certified thermometer. All calibration factors are determined on a semi-annual basis.

7.2.5 Sieves

Calibration

A sieve analysis is performed at the start of each week using a known quantity of specific materials. Percent retained on each sieve is retained in a bound notebook which is kept in the laboratory.

Sieves will be checked for accuracy of sieve opening size prior to use with NBS calibrated glass spheres, as specified in Chapter 3 of "Procedures in Sedimentary Petrology". Sieve opening size will be checked at a frequency of once per twenty samples.

Standard

All sieves are calibrated against the standard sieve sizes previously established by ASTM. Due to the potential for particle breakdown during the sieve testing procedure, a new calibration sample is utilized every three months.

7.2.6

Atterberg Limit Device

Calibration

At the start of each week a 1 cm-thick block is placed under the liquid limit cup to ensure that the cup drop is at the proper height. Measurements are recorded and entered in the bound laboratory notebook.

Standard

The liquid limit device is calibrated against the standard ASTM procedure. Set screw readjustment may be required to establish standard cup drop after a series of tests (typically every five tests). If readjustments are required at a greater frequency than every five tests, a new liquid limit apparatus will be obtained.

7.2.7

Hydrometer

Calibration

All hydrometers are placed in separate columns of distilled water to determine accuracy of the hydrometer. Measurements are recorded and entered in the bound laboratory notebook.

Standard

The hydrometers are allowed to achieve static equilibrium and readings are obtained. All readings are checked against standard readings established by the manufacturer.

7.2.8 Triaxial/CBR Press

Calibration

The press is calibrated for each new test series or five tests to determine the vertical speed of the riser plate and load cell accuracy. Measurements are recorded in the bound laboratory notebook.

Standard

The variable speed rates are checked with stop watches and dial gauges to ensure accuracy in the rate of vertical rise of the plate. These variable rates are checked against standardized rates established through ASTM.

7.2.9 Permeability Panel

Calibration

Pressure calibrations are performed before each test series to ensure that a accurate pressure head is established across the test sample. Pressure calibrations are recorded in the bound laboratory notebook.

Standard

Pressure calibrations are checked against standards established by ASTM and the manufacturer.

7.2.10 Consolidation Device

Calibration

Load frame weights are checked before each test series. Calibration weights are used to check load frame weights. All measurements are recorded in the bound laboratory notebook.

Standard

Load frame weights are checked against standards established by ASTM and the manufacturer.

7.2.11

Compaction Hammer

Calibration

Hammers are checked before each test series. The hammer drop is recorded in the bound laboratory notebook.

Standard

Hammer drop is checked against standards established by ASTM and the manufacturer.

Table 7-1 Instrumentation

Instrument/Equipment	Quantity	Make/Model Number
Standard Testing Sieves	12	Brainard-Kilman PCF Series 8 inch diameter
Sieve Shaker	1	Soil Test - CL-305A
Hydrometer Test Set	2	Brainard-Kilman - #S-305
Liquid Limit Test Set	2	Brainard-Kilman - #S-270
Compaction Test Sets	4	Brainard-Kilman - #S-320 Brainard-Kilman - #S-326 Brainard-Kilman - #S-311 Brainard-Kilman - #S-310
Mechanical Lead Frame	1	Brainard-Kilman - #S-610
Triaxial Compression/ Permeability Panel	2	Brainard-Kilman - #S-500
Triaxial Pressure Cell	1	Brainard-Kilman #S-510
Flex-Wall Permeameter	5	Brainard-Kilman - #S-480
Top Lead Balance	1	Brainard-Kilman - #GW-120
Thermometers	2	Brainard-Kilman #G-170 Brainard-Kilman #G-178
Analytical/(Electronic) Balance	1	Brainard-Kilman #GW-101
Oven	1	Brainard-Kilman #GO-220
Consolidation Frame	1	Soil Test - #C-280

Table 7-2 Calibration Table

Instrument	Initial Calibration			Continuing Calibration Verification		
	Frequency	Acceptance Criteria	Frequency	Acceptance Criteria	Corrective Action	
Sieve Analysis	At start of each test series	NBS calibrated glass beads used to calibrate sieves. See Section 7.2.5.	Every 20 tests	Percent retained must meet minimum ASTM criteria. Max variability $\leq 2\%$	Check out of spec sieve for damage. Rerun calibration. If still out of spec, replace sieve	
Atterberg Limits	At start of each week	Cup drop must meet ASTM criteria. Max 1 CM $\pm$ 1mm	Every 5 tests	Must meet ASTM criteria. Max 1 cm $\pm$ 1mm	Reset cup drop. Check set screw for wear. Replace if worn. Check base for wear (denting). If dent exceeds 1 mm in depth, replace base	
Hydrometer Analysis	At start of each week continuing calibration	Reading of 0 in column of distilled water $\pm$ 1 mm	Every 10 tests	Reading of 0 in columns of distilled water $\pm$ 1 mm	Check hydrometer for cracks or damage. Rerun calibration	
Unconfined Compression Analysis (UCG, CBR, etc.)	Each new run	Vertical speed calibration verification with $\pm$ 5% and lead cells calibrated within 5%	Every 5 tests	Same as Initial	Check dial gauge gearing for wear. If worn, replace. Check load cell for deformation and damage. If damaged or deformed, replace	
Triaxial Compression Analysis (UU, CU, CD)	Each new run	Same as unconfined compression with chamber pressure cell as an added item. Chamber pressure cell and regulator calibrated within 5%	Every 5 tests	Same as Initial	Same as unconfined compression analysis. Check chamber pressure cell and regulator for damage or wear. If damaged or worn, replace. Check chamber for leaks	
Permeability Analysis		Same as Triaxial pressure cell calibration	Every 5 tests	Same as Initial	Same as Triaxial chamber cell check.	
Consolidation Analysis		Incremental load readings within $\pm$ 5%.	Every 5 tests	Same as Initial	Check load arm hinge for wear and load weights for damage. If wear or damage is noted, replace	
Compaction Analysis		Hammer drop height within 5%.	Every 5 tests	Same as Initial	Check hammer for denting or elongation. If hammer drop criteria can't be met, replace.	
Balance	At start of each test series	Within 0.01 grams for analytical balance. Within 1.0 grams for top load balance	Every 5 tests	Same as Initial	Reset adjustment screen for proper reading on calibration weights	
Ph Meter	Each new run	Utilize various buffer solutions. Variability within $\pm$ 0.1	Every test	Same as Initial	Try new buffer solution. Recheck, if criteria still can't be met, resubmit meter to manufacturer for repair	

Organic Content Test and Bulk Density Test utilize balance equipment only.

AR300246

8.0

PREVENTATIVE MAINTENANCE

Instruments and other laboratory equipment occasionally require maintenance, either for preventative reasons or to correct malfunctions. All principal instruments are covered under service contracts with the manufacturer. The laboratory manager is responsible for scheduling any necessary maintenance with the manufacturer, whether preventative or the result of instrument malfunction.

Each piece of equipment must have a written preventative maintenance schedule as recommended by the manufacturer. Preventative maintenance includes activities such as cleaning, oiling, and routine part replacement. It is the laboratory manager's responsibility to ensure that preventative maintenance is scheduled and adhered to. This responsibility may be delegated to a qualified laboratory technician. A Preventative Maintenance Table is attached, listing the routine procedures and the frequency performed.

An equipment maintenance notebook is established on the day of installation for each piece of equipment. The front of the book lists the type of equipment, the manufacturer's name, the model number and the serial number. If desired, calibration data may be kept in the same book.

All maintenance work done on the equipment, no matter how minor, is recorded in this notebook. This includes both preventative and corrective maintenance. In the case of non-routine repairs for corrective purposes, the record documents the nature of the failure, how and when the defect was discovered, and a description of any remedial action taken. All records are kept in ink, and signed by the person doing the work or supervising the work done by an outside technician or employee of a service firm. The notebook is kept near the vicinity of the equipment and is readily available.

Whenever possible the original instrument instruction manual is kept in the laboratory with the instrument. If the manual is small and could possibly be easily lost, a copy of the manual is made for the laboratory and the original is kept with the technical director.

In the event of a major equipment failure, the laboratory may use an alternate method or instrument. If ERM is unable to perform any analysis due to instrument failure, ERM will attempt to sub-contract those analyses to a certified laboratory with the client's prior approval.

Table 8-1 Preventative Maintenance Table

Instrument	Item	Maintenance Procedure	Frequency
Sieves	Wire Mesh	Inspect, replace	As required
Atterburg Limit Device	Cup	Inspect, replace	As required
	Base unit	Inspect, replace	As required
Hydrometer	Hydrometer Bulb	Inspect, replace	As required
	Graduated Cylinder	Inspect, replace	As required
Triaxial/VCC Press	Speed switch	Inspect, replace	As required
Permeability Panel	Regulators	Inspect for obstruction or ferrule breakage	As required
	Compressor	Inspect for leaks	Daily/as required
	Water Supply	Check reservoir supply	Daily/as required
Consolidation Device	Dial Gauges	Inspect for smooth movement	As required
	Load Arms	Inspect for wear	As required
Compaction Device	Mold Collar	Inspect for wear	As required
	Mold Base	Inspect for wear	As required
	Ramming Hammer	Inspect and clean, check for wear	As required
Balances	Calibration/Accuracy	Check with Class S Weights	Daily
	Calibration	Inspect and/or clean	Annually
Ovens	Temperature Monitoring	Check and record	Daily
Refrigerators	Temperature Monitoring	Check and record	Daily

9.0

INTERNAL QUALITY ASSURANCE CHECKS

Each specific analytical method includes the particular quality control requirements performed to ensure that the data produced is of known quality. In addition to the necessary calibration standards, these quality control checks may consist of one or several different types of checks. Duplicates, reference materials and control samples are employed as appropriate for the analysis. The general criteria for use of each of these types will be outlined below.

9.1

DUPLICATE SAMPLES

Duplicate sample analysis is the analysis of the same sample twice in order to determine the precision of the analysis. The relative percent difference (RPD) between the two determinations is calculated and compared to values prescribed by EPA or determined by statistical analysis of past information generated in our laboratories. Typically, 10% of all samples will be run as duplicates. The acceptance criteria for duplicates is typically an RPD $\leq 50\%$. If this criteria is not met, the test in question will be stated with a qualifier denoting the variation.

9.2

QUALITY CONTROL CHECKS

Quality control check standards are samples which contain a known amount of a specific material (e.g., sand, silt, clay, etc.). These are analyzed in each set to ensure the accuracy of the analytical procedure. The quality control checks may be from the Association for Standards and Testing Methods (ASTM), a commercial source, or prepared in our own laboratory. Accuracy information determined from reference materials is valuable because variables specific to sample matrix are eliminated. The acceptance criteria for this type of quality control is either dictated by the agency from whom the material is obtained or by statistical analysis of past information generated in our laboratories.

Quality control check samples are samples which are analyzed in each batch to ensure the proper working of the analytical procedure. The quality control checks may be from the ASTM, a commercial source, or prepared in our own laboratory. The acceptance criteria for this type of quality control sample is either dictated by the agency from whom the material is obtained or by statistical analysis of past information generated in our laboratories.

9.3 **CONTINUING CALIBRATION STANDARDS**

Continuing calibration standards are standards which are run in the middle or at the end of test batch to ensure that the instrumentation has not drifted out of calibration. These must be run on at least a 5% frequency. If the continuing calibration standard does not fall within the acceptable range, the equipment must be checked and recalibrated prior to performing any other analyses.

9.4 **ACCEPTANCE CRITERIA/CORRECTIVE ACTION**

Due to the nature of geotechnical testing, laboratory duplicate precision often may not be met. The only means of corrective action is documentation. The results of all quality control samples are compared to the acceptance limits before data is reported. If any result is not within the acceptance criteria, corrective action suitable to the situation must be taken. This may include, but is not limited to, checking calculations, examining the results of other quality control samples analyzed with the same batch, qualifying results with a comment stating the observed deviation, and reanalysis of all the samples in the batch. The cause and solution to the problems are documented to prevent recurrence. These are used by the quality assurance department to check for trends which may indicate method bias.

These programs have been found to be invaluable in monitoring our procedures and detecting situations where the system was tending to lose control before it became serious enough to affect the integrity of the sample results.

10.0 DATA REDUCTION, VALIDATION, AND REPORTING

10.1 DATA REDUCTION

Reportable values are calculated as specified in the test method. The raw data is calculated or reduced into reportable values in three ways in the lab: manually, by an external computer program, and by the data system that collects the raw data.

The raw data is manually reduced into reportable values by the technician. His/her work is checked by the laboratory manager calculation and transcription errors. The raw data is filed for permanent storage. The worksheets are initialed by the technician and by the laboratory manager who enters the data into the computer for compiling. The worksheets are filed in the group folder for the samples. All data is identified by a batch number consisting of three parts: a test number, the data, and the technician's initials.

After the report is printed, the technical director proofreads the report against the worksheets, giving a final validation step to the process.

10.2 DATA VALIDATION

Data integrity is checked by the technicians/laboratory manager and by the technical director. The laboratory manager is responsible for checking the raw data in the workbooks, the instrument logs, calibration integrity, and calculations. The technical director also checks the calibration integrity, calculations, and internal chain-of-custody.

Data validation of laboratory quality control checks is performed by the technical director. Each time that QC samples are analyzed, the results are recorded and a copy of the batching data field is given to the technical director.

Project data is validated by the technical director prior to release of the data to the client. This review includes the chain-of-custody documentation, all laboratory sample login documents, the analytical worksheets, the quality control data, and the analytical report.

10.3 DATA REPORTING

Results are reported as specified in the test method. The technical director is responsible for the generation of all final reports. He reviews, proofreads, and signs all reports prior to their release to the client.

10.4 DATA STORAGE

All raw data, including magnetic tape and media, laboratory books, instrument printouts, and extraction logs, will be stored in climate controlled storage for at least ten years. Archived records are stored numerically by sample group and chronologically by test identification. The magnetic data has associated hard copies stored in files. All storage areas are logged and indexed.

11.0 CORRECTIVE ACTION

Quality Control (QC) samples are analyzed with each test series performed in the laboratory in order to ensure the quality of the data. One of the most effective means of preventing trouble is to respond immediately to indications of suspicious data or equipment malfunctions. Taking corrective action immediately can minimize or prevent the collection of poor quality data. When QC data fall outside established acceptance criteria, the cause of this irregularity must be investigated, corrected and documented as soon as possible. The documentation will be used to prevent a recurrence of the problem and to inform management of the situation. All QC data must be submitted to the technical director after its generation.

The laboratory has a QC notebook or file for retaining information involving QC data. The material kept in this notebook includes documentation of investigation of QC outliers. The technical director is responsible for maintaining this notebook. All entries in the notebook are initialed and dated.

The statistical limits established by the technical director based on our internal data may result in acceptance limits that are narrower than limits set by recognized standard-setting organizations (e.g., ASTM, etc.). In such cases, the limits set by the standard-setting organization will take precedence over internally generated limits.

Every attempt is made to investigate as soon as possible after notification is received. A Corrective Action Form as shown in Figure 11-1 is initiated.

11.1 STATE OR FEDERAL AGENCY CORRECTIVE ACTION

ERM, Inc., will implement corrective actions that have been submitted by a state or federal agency to ensure the quality of the data. These may be triggered by audits, by performance evaluation samples, or by split samples.

12.0 PERFORMANCE AND SYSTEM AUDITS

12.1 SYSTEM AUDITS

The technical director shall conduct quarterly audits of analyses or phases of analyses which are representative of the work routinely performed. Audits are performed to ensure that laboratory personnel are adhering to the procedures set forth in our Quality Assurance Plan. The audit results including a list of deficiencies that must be addressed to improve, modify, or correct the system are reported to the department manager and the laboratory manager. This is discussed in Section 13.0.

Inspections include direct observation of technicians as they perform the various phases of the analyses. The purpose of this type of audit is to ensure that no deviations from the written methods occur. Inspections also ensure that the written methods are followed, and may include review of notebooks, preparation data, calibrations, standardizations, maintenance of equipment, quality control records, computer programming, and personnel training records. Direct observation may be used, although traceability audits (auditing by ensuring that an accurate paper trail exists) are also useful.

At the completion of each audit, a review of any significant problems uncovered during the audit will be held with the laboratory manager. The findings of each audit will be reported in writing to management by the auditor. Internal Quality Control samples are submitted to the laboratory periodically to document the performance of the laboratory or individual technician. These samples are obtained from an external source of quality control samples. This process is discussed in Section 9.0.

External audits are performed by outside agencies and entities such as state and federal agencies. ERM's policy is to welcome such audits as they are necessary for certification or other reasons. Full cooperation will be given to auditing personnel prior to and during audits.

1.2

PERFORMANCE AUDITS

Performance audits are performed quarterly to determine the accuracy of the laboratory. These audits are performed by the technical director and involve the use of samples which have quantities which are not known to the technicians. They may include performance evaluation samples, split samples, quality control samples or check samples.

All deficiencies determined during a performance audit must be addressed by the laboratory manager. This response is a written response to the technical director and it is filed in the QA file.

13.0 QUALITY ASSURANCE REPORTS

The technical director is responsible for preparing periodic quality assurance reports which will indicate the performance of the laboratory and the overall data quality. These reports include a summary of data accuracy, precision, and completeness, a summary of performance and laboratory audits, and an outline of any quality assurance issues and resolutions, as well as any major modifications resulting from corrective actions. The reports are distributed to the department manager and the laboratory manager.

Reports resulting from department and traceability audits or corrective actions are addressed promptly in order to resolve possible problems immediately. These reports are given to the laboratory manager. Depending on the magnitude of the issues addressed, these reports may or may not be summarized in the Laboratory Quality Assurance Report.

14.0

RESUMES

Attachment 3
pH Meter Manufacturer's
Instructions

AR300258

Replacement	\$ 27.50
High Temp	83.00
Spear Type Probe Unit, 30" lead	13.00
High pH Probe Unit, 30" lead	38.00
pH Probe Unit with 10 ft. lead in 3/4" nipple	33.00
Carrying Case	30.00
Buffer Kit, 500 ml	9.00
Buffer Salts, 6 env. per pkg. of one pH value	6.00
Buffer Solution, per pint	3.39
9 Volt Battery	1.10

This instrument is unconditionally guaranteed for one year from date of purchase except for broken or desensitized Probe Units. The guarantee is void if the instrument has been dropped, severely jarred, or taken apart.

If the instrument requires service, we recommend that it be floated in crushed newspaper and shipped prepaid to:

Analytical Measurements, Inc.
31 Willow Street
Chatham, New Jersey 07928

Prices subject to change without notice.

AR300259



Analytical Measurements

Telephone: (201) 273-7500

31 WILLOW STREET CHATHAM NEW JERSEY 07020



Printed in U.S.A.

Operating Instructions

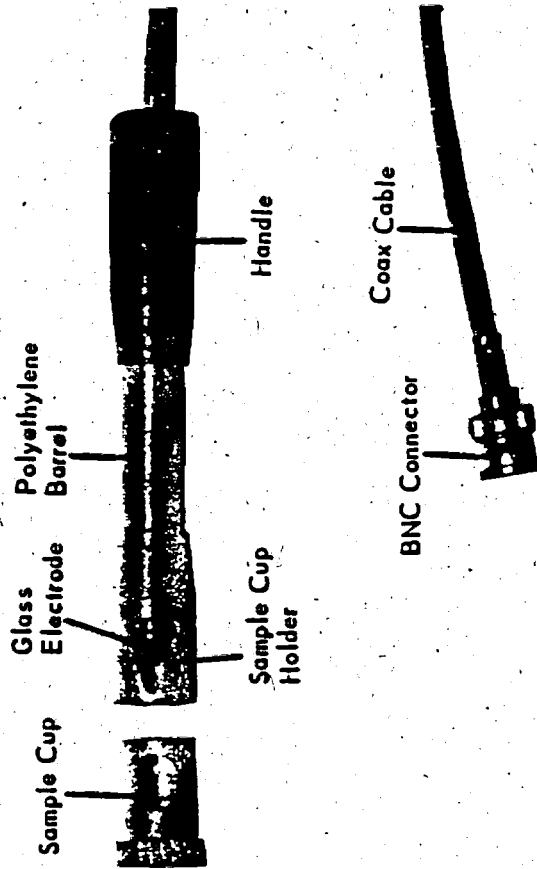
This Probe Unit is of a new type requiring no refilling with KCl solution. This is accomplished with gel filling and a ceramic junction. The Probe Unit must be immersed above the top of the cup holder in order to make connection with the ceramic junction in the body of the electrode.

1. Set "Selector" Switch to "0" position. Rotate "Calibrate" knob clockwise and counterclockwise and observe pointer movement. Pointer should move freely up and down scale as knob is rotated. Then set pointer to 7 pH with "Calibrate" knob.
2. Remove sample cup from Probe and rinse Probe in distilled or deionized water. (If neither is available then rinse with tap water.)
3. Immerse Probe Unit in a beaker of 7 pH buffer solution. Turn Selector Switch to pH. Rotate Calibrate knob until pointer reads 7 pH.
4. Rinse Probe Unit under tap water or in a vessel of clean water, and immerse into a beaker of 4 pH buffer solution. The meter should now read 4 pH.
5. Rinse Probe Unit and instrument is now ready to test required samples.
6. Turn Selector Switch to 0 position and note reading. This is important. (If Calibrate knob is inadvertently rotated during later testing, it would not be necessary to go through Steps 3 and 4 again.) Reset pointer by means of Calibrate knob to value noted above with Selector Switch in 0 position.
7. Turn Selector Switch to pH. Set Temperature dial to temperature of sample under test.

When finished with samples, rinse Probe, replace sample cup and fill sample cup with water to keep bulb moist.

Turn Selector Switch to "Off".

To replace battery, remove back of meter (4 corner screws). Remove old battery, snap on new battery, place in clip and replace back. Use a standard 9 volt Transistor Radio battery available locally.

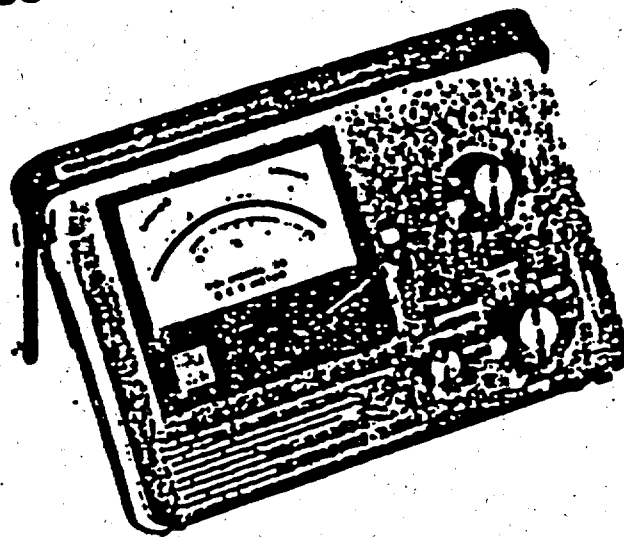


AR300260

Attachment 4
Specific Conductance Meter
Manufacturer's Instructions

AR300261

**INSTRUCTIONS FOR YSI MODEL
33 AND 33M S-C-T METERS**



**Scientific Division
Yellow Springs Instrument Co., Inc.
Yellow Springs, Ohio 45387 • Phone 513-767-7241**

PRICE INCLUDING HANDLING \$5.00

AR300262

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GENERAL DESCRIPTION

The YSI Model 33 and 33M S-C-T Meters are portable, battery powered, transistorized instruments designed to accurately measure salinity, conductivity and temperature. They use a probe consisting of a rugged plastic conductivity cell and a precision YSI thermistor temperature sensor combined in a single unit.

Conductivity with the Model 33 is expressed as micromhos/cm-meter ($\mu\text{mhos/cm}$); with the 33M, it's millimhos/meter (mS/m). These are measurements of the electrical conductance the sample would show if measured between opposite faces of a 1cm cube. (Conversion information: $1 \mu\text{mho/cm} = 0.1 \text{ mS/m}$) Salinity is the number of grams of salt/gram of sample (the = parts per thousand). This measurement assumes the sample contains a "standard" sea water salt mixture. The sample temperature is measured in degrees Celsius.

Salinity measurements are manually temperature compensated by direct dial. Conductivity measurements are not temperature compensated. However, a temperature function is provided on the instrument to aid with calculation of corrections. Also, when just temperature and conductivity are known it is possible to calculate salinity, and when only temperature and salinity are known it is possible to calculate conductivity.

SPECIFICATIONS

Model 33 Conductivity

Ranges

0-500, 0-5,000, 0-50,000 $\mu\text{mhos/cm}$ with YSI 3300 Series Probes (Note: The "umbo" designation on the meter are a shorthand form for " $\mu\text{mho/cm}$ ")

Accuracy

$\pm 2.5\%$ max error at 500, 5,000 and 50,000 plus probe
 $\pm 3.0\%$ max error at 250, 2,500 and 25,000 plus probe.
 See Error Section.

2

Readability

25 $\mu\text{mhos/cm}$ on 500 $\mu\text{mho/cm}$ range
 25 $\mu\text{mhos/cm}$ on 5,000 $\mu\text{mho/cm}$ range
 250 $\mu\text{mhos/cm}$ on 50,000 $\mu\text{mho/cm}$ range

Temperature Compensation

None

Model 33M Conductivity

Ranges

0-50, 0-500, 0-5,000 mS/m with YSI 3300 Series Probes
 $\pm 2.5\%$ max error at 50, 500 and 5,000 plus probe
 $\pm 3.0\%$ max error at 25, 250 and 2,500 plus probe
 See Error Section.

Accuracy

Readability

0.25 mS/m on 50 mS/m range
 2.5 mS/m on 500 mS/m range
 25.0 mS/m on 5,000 mS/m range

Temperature Compensation

None

Salinity

Range:

0-40 ‰ in temperature range of -2 to +45°C

Accuracy

Above 4°C, $\pm 0.9\%$ at 40 ‰; and $\pm 0.7\%$ at 20 ‰ plus conductivity probe
 Below 4°C, $\pm 1.1\%$ at 40 ‰; and $\pm 0.9\%$ at 20 ‰ plus conductivity probe
 See Error Section

Readability

0.2 ‰ on 0-40 ‰ range
 Manual by direct dial from -2 to +45°C

3

Temperature

Range

-2 to +50°C
±0.1°C at -2°C, ±0.6°C at 45°C

Accuracy

plus probe

See Error Section

±0.15°C at -2°C to ±0.37°C at 45°C

Readability

Power Supply

Two D-size alkaline batteries. Everyday E95 or equivalent provides approximately 200 hrs of operation.

YSI 3300 Series Conductivity/Temperature Probe

Probe

Nominal Probe Constant, $K = 5/cm$

±2% of reading for conductivity and

salinity

Error of ±0.1°C at 0°C and

±0.3°C at 40°C

Accuracy

Instrument

Ambient Range

Satisfactory operation -5 to +45°C.
A maximum error of ±0.1% of the reading per °C change in instrument temperature can occur. This error is negligible if the instrument is readjusted to redline for each reading.

OPERATION PROCEDURE

1. Setup

(a) Adjust meter zero (if necessary) by turning the bakelite screw on the meter face so that the meter needle coincides with the zero on the conductivity scale.

(b) Calibrate the meter by turning the MODE control to REDLINE and adjusting the REDLINE control so the meter

needle lines up with the redline on the meter face. If this cannot be accomplished, replace the batteries.
(c) Plug the probe into the probe jack on the side of the instrument.

(d) Put the probe in the solution to be measured (See Probe Use).

2. Temperature

Set the MODE control to TEMPERATURE. Read the temperature on the bottom scale of the meter in degrees Celsius. Allow time for the probe temperature to come to equilibrium with that of the water before reading.

3. Salinity

(a) Transfer the temperature reading from Step 2 to the °C scale on the instrument.

(b) Switch the MODE control to the SALINITY position and read salinity on the red 0-40‰ meter range.

(c) Depress the CELL TEST button. The meter reading should fall less than 2%. If greater, the probe is fouled and the measurement is in error. Clean the probe and re-measure.

Conductivity on Model 33 (Model 33M data are in parentheses.)

(a) Switch the MODE control to the X100 scale if the reading is below 50 on the 0-500 range (5.0 on the 0-50 range). Switch to the X10 scale if the reading is still below 50 (5.0) switch to the X1 scale. Read the meter scale and multiply the reading appropriately. The answer is expressed in $\mu\text{mhos/cm}$ (mS/m). Measurements are not temperature compensated.

Example: Meter Reading 247 (24.7)

Scale X10

Answer 2470 $\mu\text{mhos/cm}$

(247.0 mS/m)

(b) When measuring on the X100 and X10 scales, depress the CELL TEST button. The meter reading should fall less than 2%, if greater, the probe is fouled and the measurement is in error. Clean the probe and re-measure.

NOTE: The CELL TEST does not function on the X1 scale.

5. Error

The maximum error in a reading can be calculated by using the graphs in the following sections.

(1) Temperature

The temperature scale is designed to give the minimum salinity error when the temperature readings are used to compensate salinity measurements.

Figure 1 shows total error for probe and instrument versus °C meter reading.

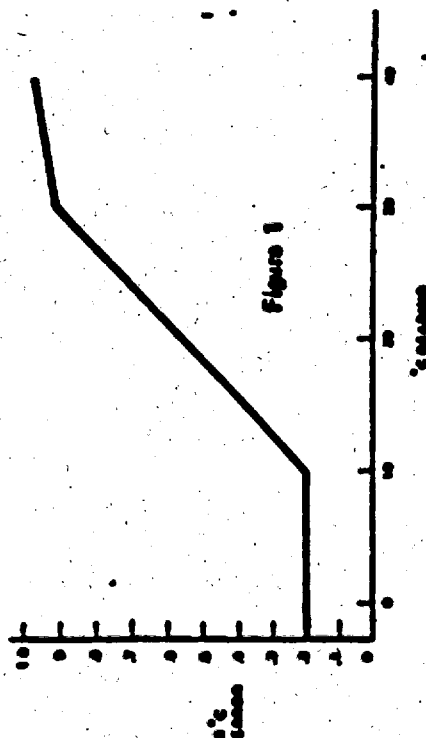


Figure 1

Example: Meter Reading 15°C
Total Error 0.4°C

Accuracy: 15°C ± 0.4°C for probe and instrument combined

(2) Conductivity on Model 33 (Model 33M data are in parentheses)

Figure 2 shows the worst-case conductivity error as a function of the conductivity reading for the probe and instrument combined



Figure 2

Example: Meter Reading 360 µmhos/cm (36 mS/m)

Scale X10

% Reading Error: ± 4.5%

Accuracy: 3600 ± 162 µmhos/cm

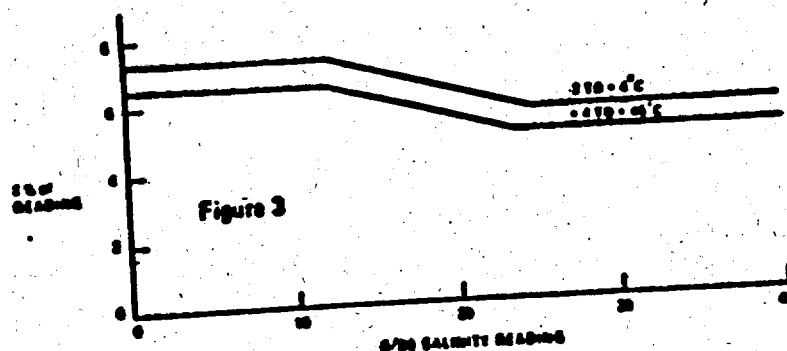
(360 ± 16.2 mS/m)

for probe and instrument

7

(3) Salinity

The salinity readings are a function of temperature and conductivity, therefore the accuracy is a function of both. The temperature scale and temperature control have been designed to minimize the temperature error contribution to the salinity error. The error shown in Figure 3 is the total of the temperature and conductivity probe, the temperature scale and the salinity scale error.



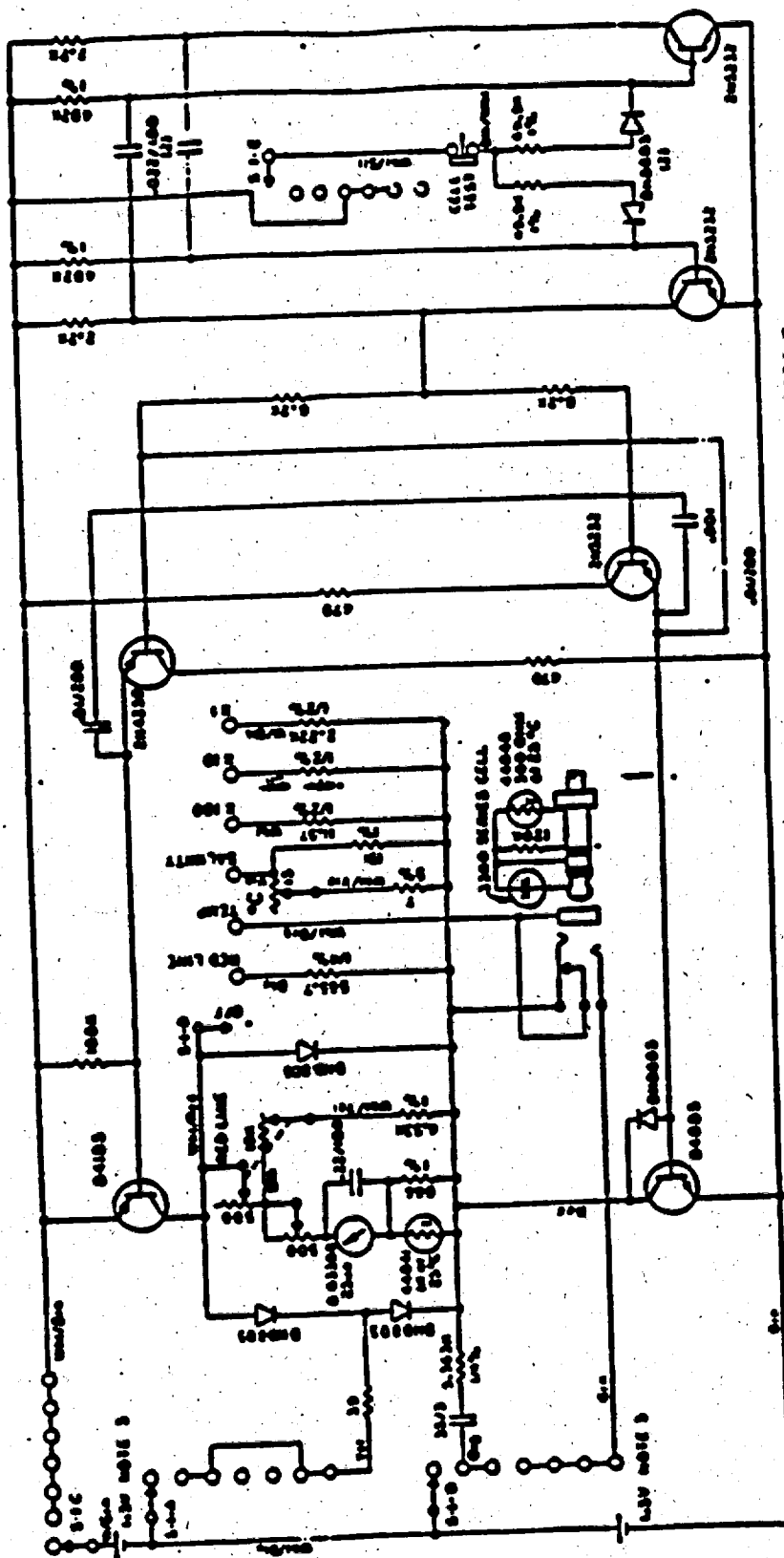
Example: Meter Reading: 100/00. @ 10°C

% of
Reading
Error:

6.5%

Accuracy:

10% ± 0.65% for all
errors, combined worst
case.



YSI MODEL 33 AND 33M B-03321-F

- NOTE
1. Resistor values in ohms are 1000 ohms
 2. "K" indicates thousands of ohms
 3. "M" indicates millions of ohms
 4. The value shown on the instrument may differ from that on the instrument of 100 ohms value
 5. Not to be used for reference purposes
 6. Battery is 1.5V
 7. Not to be used

AR300268

CIRCUIT DESCRIPTION, MAINTENANCE AND CALIBRATION

1. Description

The circuit is composed of two parts, a multivibrator and switching transistors. The multivibrator produces a square waveform voltage. The square wave is applied to two switching transistors. They alternately apply two batteries of opposite polarity to the probe thus providing AC power which minimizes polarization effects. The meter is in series with one battery and measures the current from it. The current from the battery is proportional to the conductance of the cell. Salinity is measured in a special range conductivity circuit which includes a user-adjusted temperature compensator. In the temperature, redline and X1 positions the multivibrator operates at 100 Hz. In the salinity, X100 and X10 positions the multivibrator operates at 600 Hz and in these ranges pushing the CELL TEST button drops the frequency to 100 Hz allowing the operator to judge the degree of probe polarization.

2. Maintenance

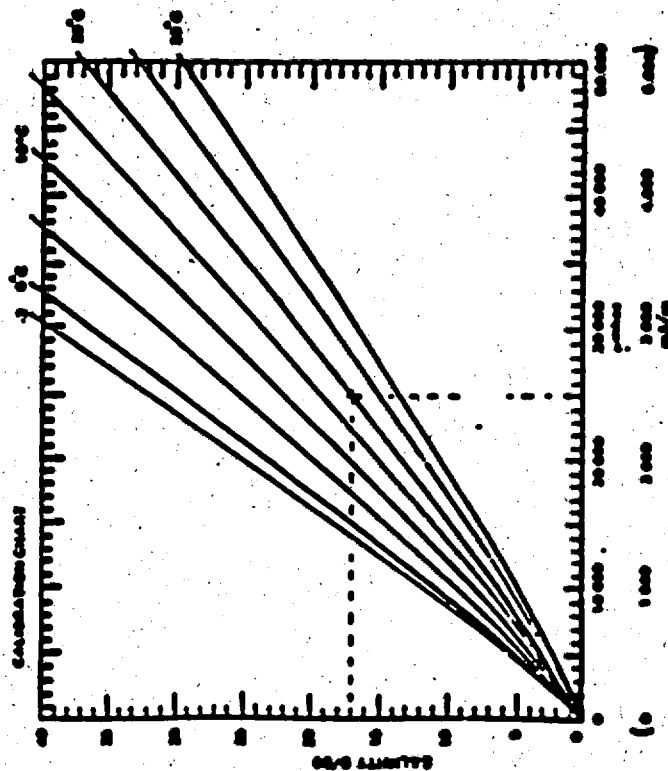
The only maintenance required is battery replacement. Two "D" size alkaline flashlight cells, such as Eveready E95 or equivalent, will provide 200 hrs of operation. Accuracy will not be maintained if zinc-carbon "D" cells are used. Battery replacement is indicated when the redline adjustment cannot be accomplished.

Replace batteries every six months to reduce the danger of corrosion due to leaky batteries. To replace batteries, remove the six screws from the rear plate. The battery holders are color coded. The Positive (+ button) end must go on red.

3. Calibration of Model 33 (Model 33M data are in parentheses.)

It is possible for the temperature knob to become loose or slip from its normal position. In an emergency the dial can be re-positioned. It must be emphasized that this is an emergency procedure only, and that the instrument should be returned to the factory for proper recalibration at the earliest opportunity.

- (a) Read the temperature and conductivity of the solution. Determine the salinity of the solution by running a line vertically on the graph from this conductance value until it intersects the appropriate °C line (interpolate as required for temperature between the given °C lines) from this intersection extend a



10

line horizontally to the edge of the graph. This determines the salinity for this sample.

Example: 25,000 μ mhos/cm and 20°C gives a salinity of 17 (Example: 2,500 mS/m and 20°C gives a salinity of 17).

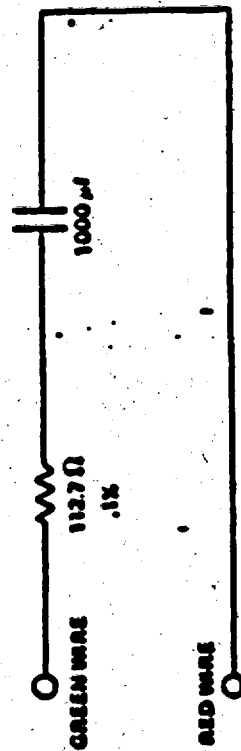
- (b) Remove the °C knob, switch to SALINITY, and turn the control shaft until the meter needle indicates the salinity value determined in Step (a). In the example given, the value is 17.

- (c) Switch to TEMPERATURE. (Note: This temperature reading must be the same as Step (a), if not, begin again at Step (a).) Place the knob on the control shaft (without turning the control shaft) with the knob pointer at the same temperature as the meter reading and tighten both set screws securely.

At earliest opportunity recalibrate using the following procedure or return the instrument to factory for service.

- (a) Set the instrument for a salinity measurement as normal.
(b) Substitute a 1000 μ f capacitor and 112.7 ohm 0.1% tolerance resistor for the probe.

Connect the resistor and capacitor between the green wire and red wire on the jack connections inside the instrument.



11

AR300270

11 17 70 P

(c) Turn the temperature dial until the meter reads redline. Now insert the temperature knob with the arrow at 25°C. This is a temporary calibration only. Return the instrument to the factory for proper recalibration.

PROBE

1. Description of YSI 3300 Series Conductivity/Temperature Probe

The YSI 3300 Series Conductivity Probes are designed for field use, embodying construction and design for rugged, accurate service. Each probe features a built-in cell constant of 5.0 (500 Q/M) $\pm 2\%$, a precision YSI thermistor temperature sensor of $\pm 0.1^\circ\text{C}$ accuracy at 0°C and $\pm 0.3^\circ\text{C}$ at 40°C , and a low capacitance cable assembly terminating in a three terminal 0.25" dia. phone type connector.

The 3310 has a 10 ft. cable and the 3311 is a 50 ft. version. Other lengths are available on special order.

The probe has a rigid P.V.C. body plated pure nickel electrodes, and a durable cable providing resistance to a wide range of water-borne substances.

2. Maintenance

(a) Cleaning

When the cell test indicates low readings the probable cause is dirty electrodes. Hard water deposits, oils and organic matter are the most likely contaminants.

For convenient normal cleaning soak the electrodes for 5 minutes with a locally available bathroom tile cleaning preparation such as: Dow Chemical Bathroom Cleaner, Harsco Industries' Ruffy, Tile, Porcelain, and Chrome Cleaner, Johnson Wax' Envy, Instant Cleaner, or Lycol Brand "Bath Tub, Tile Cleaner".

For stronger cleaning a 5 minute soak in a solution made of 10 parts distilled water, 10 parts isopropyl alcohol and 1 part HCl can be used. Always rinse the probe after cleaning and before storage.

CAUTION Do not touch the electrodes inside the probe.

Platinum black is soft and can be scraped off.

If cleaning does not restore the probe performance, re-platinizing is required.

(b) Re-Platinizing

Equipment Required --

- (1) YSI 33140 Platinizing Solution, 2 M or (3% platinum chloride dissolved in 0.025% lead acetate solution)
- (2) YSI Model 33 or 33M S-C-T Meter
- (3) 50 ml glass beaker or equivalent bottle
- (4) Distilled water

Procedure --

- (1) Clean the probe as in Section (a) -- either method
- (2) Place the cell in the beaker and add sufficient YSI 33140 solution to cover the electrodes. Do not cover the top of the probe.
- (3) Plug the probe into the Model 33 or 33M, switch to the X100 scale to platinize the electrode. Move the probe slightly to obtain the highest meter reading and continue platinizing for the approximate time shown below.

Meter Reading		Time (minutes)
mmhos/cm	mS/m	
30,000	3,000	5
25,000	2,500	8
20,000	2,000	8
15,000	1,500	11
10,000	1,000	16

- (4) After the elapsed time remove the probe and rinse in fresh water.
- (5) Return the solution to its container. 2 oz. of solution should be sufficient for 50 treatments.

(c) Storage:

It is best to store conductivity cells in deionized water. Cells stored in water require less frequent platinization. Any cell that has been stored dry should be soaked in deionized water for 24 hours before use.

3. Probe Use

- (a) Obstructions near the probe can disturb readings. At least two inches of clearance must be allowed from non-metallic underwater objects. Metallic objects such as piers or weights should be kept at least 6 inches from the probe.
- (b) Weights are attached to the cable of the YSI 3310 and 3311 Probes. The YSI 3327 Weights are supplied in pairs with a total weight of 4 ounces per pair. Should a become necessary to add more weight to overcome water currents, we suggest limiting the total weight to two pounds (9 pairs). For weights in excess of two pounds use an independent suspension cable. In either case, weights must be kept at least 6 inches away from the probe.
- (c) Gentle agitation by raising and lowering the probe several times during a measurement insures flow of specimen solution through the probe and improves the time response of the temperature sensor.

4. Cell Calibration & Standard Solutions

The YSI 3300 Series Cells are calibrated to absolute accuracy of $\pm 1.5\%$ based on a standard solution. Since the literature on conductivity does not indicate a consistently accepted standardization method, we have chosen the 0.1 normal KCl solution method as determined by Jones and Bradshaw in 1937 as our standard. Recent textbooks, as well as the ASTM standards, concur with this choice.

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The solution is prepared by dissolving 0.745 grams of pure dry KCl with distilled water until the solution is 1 kilogram. The table below shows the values of conductivity this solution would have if the distilled water were non-conductive. However, since even high purity distilled water is slightly conductive, the measured conductivity will be higher by an amount equal to the water's conductivity.

Temperature °C	µmhos/cm	Conductivity mS/m
15	1141.5	114.2
16	1167.5	116.8
17	1193.6	119.4
18	1219.9	122.0
19	1246.4	124.6
20	1273.0	127.3
21	1299.7	130.0
22	1326.6	132.7
23	1353.6	135.4
24	1380.8	138.1
25	1408.1	140.8
26	1435.5	143.7
27	1463.2	146.3
28	1490.9	149.1
29	1518.7	151.9
30	1546.7	154.7

The operator may use the standard solution and the table to check accuracy of a cell's constant or to determine an unknown constant. The formula is shown below.

$$K = \frac{R(C + C_1)}{10^6} \quad \text{or} \quad \frac{R(S + S_1)}{10^6}$$

where K = Cell constant
 R = Measured resistance in Ω

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