WARZYN

PROJECT
10009721

US EPA RECORDS CENTER REGION 5
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REMEDIAL DESIGN/REMEDIAL ACTION QUALITY ASSURANCE PROJECT PLAN

WINNEBAGO RECLAMATION LANDFILL SITE (PAGEL'S PIT)
WINNEBAGO COUNTY, ILLINOIS

SEPTEMBER 1993

PREPARED FOR:
WINNEBAGO RECLAMATION SERVICES, INC.
ROCKFORD, ILLINOIS

• • •

PREPARED BY:
WARZYN INC.
MADISON, WISCONSIN

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APPROVALS:	DATE:
Winnebago Reclamation Landfill Site Project Manager	
Warzyn Project Manager	
Warzyn Quality Assurance Officer	
U.S. EPA, Region V, Remedial Project Manager	
U.S. EPA, Region V, Quality Assurance Manager	
IEA Laboratory Manager	
IEA Laboratory Quality Assurance Manager	

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LIST OF ACRONYMS AND ABBREVIATIONS

ARAR	Applicable or Relevant and Appropriate Requirement
ASTM	American Standards for Testing Materials
CDO	Central District Office
CLP	Contract Laboratory Program
CRL	Central Regional Laboratory
DL	Detection Limit
DQO	Data Quality Objective
GC	Gas Chromatograph
GC/MS	Gas Chromatograph and Mass Spectrometer
HRS	Hazard Ranking System
HASP	Site-Specific Health and Safety Plan
ICP	Inductively Coupled Plasma Spectrometer
IDL	Instrument Detection Limit
IEPA	Illinois Environmental Protection Agency
ITL	Inorganic Target List
MS/MSD	Matrix Spike/Matrix Spike Duplicate
NPL	National Priorities List
OTL	Organic Target List
PRP	Potentially Responsible Party
QA	Quality Assurance
QAO	Quality Assurance Officer
QAPjP	Quality Assurance Project Plan
QAS	Quality Assurance Section
QC	Quality Control
%R	Percent Recovery
RD/RA	Remedial Design/Remedial Action
RDL	Required Detection Limit
RPD	Relative Percent Difference
RPM	Remedial Project Manager
Site	Winnebago Reclamation Landfill Site
SOP	Standard Operating Procedure
SP	Sampling Plan for Field Activities
TIC	Tentatively Identified Compound
U.S. EPA	United States Environmental Protection Agency, Region V
VOA	Volatile Organic Analysis
VOC	Volatile Organic Compound
Warzyn	Warzyn Inc.
WP	Component Work Plan
WRL	Winnebago Reclamation Landfill
WRS	Winnebago Reclamation Services, Inc.

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INTRODUCTION

The United States Environmental Protection Agency (U.S. EPA) requires that all environmental monitoring and measurement efforts mandated or supported by the U.S. EPA participate in a centrally managed quality assurance (QA) program.

Any party generating data under this program has the responsibility to implement minimum procedures to assure that precision, accuracy, completeness, and representativeness of its data are known and documented. To ensure the responsibility is met uniformly, each party must prepare a written QA Project Plan (QAPjP) covering each project it is to perform.

This QAPjP presents the organization, objectives, functional activities, and specific Quality Assurance (QA) and Quality Control (QC) activities associated with the Remedial Design/Remedial Action (RD/RA) for the Winnebago Reclamation Landfill (WRL) Site.

This QAPjP is written to cover the currently defined and scheduled predesign, design, and remedial action activities, and describes the specific protocols which will be followed for sampling, sample handling and storage, chain-of-custody, and laboratory and field analysis relative to the defined activities.

The WRL is an active landfill with approximately 5 to 7 years of capacity remaining at the present fill rate. The RD/RA actions required by the Record of Decision, Consent Decree, and Statement of Work consist of multiple RD/RA activities that will be implemented in a phased approach, consistent with ongoing fill and closure activities at the landfill and U.S. EPA approved RD/RA schedules. The required RD/RA activities have been grouped into three basic components:

- Groundwater Extraction and Treatment
- Leachate and Landfill Gas Management
- Final Cover System

An RD Work Plan (WP) will be prepared for each of the above RD/RA components and will include a component specific schedule, planned predesign investigation activities, commitments for reports and submissions, and other plans required to execute the remedial design. These component RD WPs will be supported by this comprehensive QAPjP and the general RD/RA Health and

Safety Plan (HASP), but may include specific QAPjP and HASP requirements not presently identified in this QAPjP. This QAPjP along with any specific additions and/or modifications specified in the component RD WPs will provide the full documentation to assure the precision, accuracy, completeness, and representativeness of data collected to support the designs that are known and documented. QAPjP provisions for U.S. EPA required and approved compliance monitoring will be referenced to this QAPjP and modified, as required, in the respective component RDs and QAPjP amendments.

All QA/QC procedures will be in accordance with applicable professional technical standards, U.S. EPA requirements, government regulations and guidelines, and specific project goals and requirements. This QAPjP has been prepared by Warzyn Inc. (Warzyn) in accordance with the following U.S. EPA QAPjP guidance documents:

- U.S. EPA, December 1980, Interim Guidelines and Specifications for Preparing Quality Assurance Project Plans, QAMS-005/80
- U.S. EPA, January 1989, Region V Content Requirements for Quality Assurance Project Plan
- U.S. EPA, May 1991, Region V Model Superfund Quality Assurance Project Plan

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PROJECT DESCRIPTION

1.1 SITE LOCATION AND BACKGROUND INFORMATION

The Winnebago Reclamation Landfill Site (WRL Site or Site), also known as Pagel's Pit, is an active solid waste landfill licensed by the state of Illinois. The WRL Site is located in south central Winnebago County in north central Illinois, approximately five miles south of the city of Rockford, in a predominantly rural unincorporated area. The WRL Site is comprised of approximately 60 acres of land in the east central portion of Section 36, T43N, R1E and the west central portion of Section 31, T43N, R2E. The WRL Site is bounded on the west by Killbuck Creek, on the east side by Lindenwood Road. Killbuck Creek is a perennial stream that flows within 250 ft of the western WRL boundary and merges with the Kishwaukee River about two miles to the north.

The Site is located on a topographic high between Killbuck Creek and unnamed intermittent streams to the north and south. Surface topography of the Site consists primarily of an area of high relief resulting from the landfill waste disposal operations. The topography surrounding the landfill area is relatively flat to gently rolling. The ground surface ranges from elevation 790 ft mean sea level (MSL) on top of the landfill to 708 ft MSL in the floodplain of Killbuck Creek. A small leachate collection pond is located on top of the landfill.

Land use around the WRL Site is a mix of industrial, agriculture, commercial and rural residential. East of the WRL Site is the former Acme Solvent Reclaiming Inc. (Acme Solvent) site, an industrial waste disposal site which operated from 1960 to 1973.

The Acme Solvent site, an approximately 20 acre parcel, was used for the disposal of drummed wastes into unlined lagoons and drum stockpiling. The wastes disposed of at the Acme Solvent site are generally undocumented, but are known to have included solvent still-bottom sludges, nonrecoverable solvents, paints, and oils. The Illinois Environmental Protection Agency (IEPA) indicates that four

lagoons were actively used for the disposal of wastes at the Acme Solvent site, and that 10,000 to 15,000 drums may have been present at the site when it closed.

The Rockford Skeet Club is located across Lindenwood Road to the northeast. A septic tank pumping business is located to the west, a private hunting club to the southwest, and a limestone quarry to the east of the WRL Site. There are scattered residences within 1/2 mile of the Site to the north, south, southeast, and southwest.

1.2 PAST DATA COLLECTION ACTIVITY/CURRENT STATUS

A Remedial Investigation (RI) was previously conducted at the WRL Site. Additional information concerning Site history and background are presented in the RI Report dated March 1991.

1.3 PROJECT OBJECTIVES AND SCOPE

The selected remedial action includes the following major elements:

- Identification of the extent of groundwater contamination
- Groundwater extraction and treatment system
- Leachate management system
- Landfill gas management system
- Final cover system
- Control of air emissions
- Groundwater, soil gas, leachate, air, and water supply monitoring
- Deed and access restrictions
- Alternate water supply

1.4 SAMPLE NETWORK DESIGN AND RATIONALE

The sample design and rationale for sample locations will be described in detail in the Sampling Plan for Field Activities (SP) to be developed as a part of each stage of the component RDs. A general SP is included as Appendix A of this QAPjP to provide standard procedures for anticipated sampling activities.

1.5 PARAMETERS TO BE TESTED AND FREQUENCY

Parameters to be tested will be listed in the component RD SP. Sample matrices, analytical parameters, and frequencies of sample collection can be found in Table 1-1. A summary of sample volume, bottle, preservation, and packaging requirements is provided in Table 1-2. Tables will be modified and expanded as needed in the component RD SPs and QAPjP amendments to cover component-specific sample analysis.

1.6 DATA QUALITY OBJECTIVES

Data Quality Objectives (DQOs) are qualitative and quantitative statements which specify the quality of the data required to support decisions made during the RD/RA activities, and are based on the end uses of the data to be collected. As such, different data uses may require different levels of data quality. There are five analytical levels which address various data uses and the QA/QC effort and methods required to achieve the desired level of quality. These levels are:

- Screening (DQO Level 1): This provides the lowest data quality but the most rapid results. It is often used for health and safety monitoring at the site, preliminary comparison to Applicable or Relevant and Appropriate Requirements (ARARs), initial site characterization to locate areas for subsequent and more accurate analyses, and for engineering screening of alternatives (bench-scale tests). For the Site, data generated under DQO Level 1 are for health and safety monitoring. Health and Safety Monitoring activities are described in the Site-Specific Health and Safety Plan (HASP) and are not covered in this QAPjP.

- Field Analyses (DQO Level 2): This provides rapid results and better quality than in Level 1. This level may include mobile laboratory generated data depending on the level of quality control exercised. For the Site, data generated under DQO Level 2 are the purgeable VOCs in groundwater while drilling to determine screen placement.

- Engineering (DQO Level 3): This provides an intermediate level of data quality and is used for site characterization. Engineering analyses may include mobile laboratory generated data and analytical laboratory methods with full quality control documentation, with built-in QA/QC,

including calibration runs, surrogate standards, etc., and external QA/QC such as duplicate samples, field blanks, etc. Data validation will not be performed. For the site data generated under DQO Level 3 are the grain size analysis for soils.

- Confirmational (DQO Level 4): This provides the highest level of data quality and is used for purposes of risk assessment, evaluation of remedial alternatives, and Potentially Responsible Party (PRP) determination. These analyses require Contract Laboratory Program (CLP) analytical and data validation procedures in accordance with U.S. EPA recognized protocol for the site, data generated under DQO Level 4 are the TCL VOCs and SVOCs, TAL selected metals, and cyanide and the indicator parameters for groundwater samples.

- Non-Standard (DQO Level 5): This refers to analyses by non-standard protocols, for example, when exacting detection limits or analysis of an unusual chemical compound is required. These analyses often require method development or adaptation. The level of quality control is usually similar to DQO Level 4 data. These analyses require full CLP analytical and data validation procedures in accordance with U.S. EPA recognized protocol. DQO Level 5 data is not anticipated for the Site.

A summary of data generating activities, intended data uses and associated DQOs for the Site are presented in Table 1-3 which will be modified as needed in the component RD WPs and QAPjP amendments.

1.7 PROJECT SCHEDULE

A summary schedule for RD/RA activities at the WRL Site is presented as Figure 3 of the RD/RA Work Plan. A schedule of activities specific to the component RD/RAs will be included in the component RD Work Plans and RDs.

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PROJECT ORGANIZATION AND RESPONSIBILITY

At the direction of the Class A Setting Defendant, Winnebago Reclamation Services, Inc., who entered into a Consent Decree with the U.S. EPA, effective February 11, 1993 (lodged), Warzyn will serve as the Supervising Contractor to WRS for implementation of the RD/RA activities as specified in the ROD, Consent Decree, and Statement of Work. Warzyn is also tasked to prepare the RD for the groundwater extraction and treatment system. The various quality assurance and management responsibilities of key project personnel are defined below. Refer to Figure 2-1 and 2-2 for the project organizational charts. The organization structure and responsible individuals for the various RD/RA activities will be specified in the component RD Work Plans.

2.1 OVERALL RESPONSIBILITY

2.1.1 U.S. EPA Remedial Project Manager

The U.S. EPA is the lead agency and is responsible for providing oversight of the WRL Site RD/RA. The U.S. EPA Region V Quality Assurance Manager has the responsibility to review and approve the QAPjP. The U.S. EPA Remedial Project Manager (RPM) is Mr. Bernard Schorle. His responsibilities encompass acting as the coordinator of communications between the U.S. EPA and WRS, and assuring compliance with the Consent Decree and approved planning documents.

2.1.2 U.S. EPA Quality Assurance Manager

The U.S. EPA Region V Quality Assurance Manager has the responsibility to review and approve all Quality Assurance Project Plans.

2.1.3 IEPA Remedial Project Manager

The IEPA Remedial Project Manager is Mr. Fred Nika. The IEPA Remedial Project Manager will be involved in the project as agreed between U.S. EPA and IEPA.

2.1.4 WRS Project Coordinator

The WRS Project Coordinator is Mr. Gary Marzorati. He is responsible for implementing the project, and has the authority to commit the resources necessary to meet project objectives and requirements. The Project Coordinator's primary function is to ensure that technical, financial, and scheduling objectives are achieved successfully. He or his designee will report directly to the U.S. EPA RPM and will provide the major point of contact and control for matters concerning the project.

2.1.5 Warzyn Project Manager

Warzyn, as the Supervising Contractor to WRS, is responsible to WRS for the development of the RD/RA Work Plan and planning documents (QAPjP and HASP) for implementing the RD/RA activities at the WRL Site. Warzyn has also been tasked and is responsible to WRS for the development of the Groundwater RD Work Plan, implementation of predesign studies to define the extent of groundwater contamination at the west end of the WRL, and the preparation of the RD for the groundwater extraction and treatment system. Warzyn will also provide review and oversight of the preparation of component RD Work Plans and designs for the other remedial design components, as required by WRS. The Warzyn Project Manager is Mr. Alan Schmidt. The Warzyn Project Manager is responsible for providing support to WRS for the execution and completion of assigned work in compliance with requirements, objectives, and quality standards specified in the Consent Decree, Statement of Work, and approved Work Plans and planning documents. In addition he is responsible for technical quality control and oversight of assigned project elements.

The WRS Project Coordinator and Warzyn Project Manager will:

- Define project objectives and develop a detailed work plan schedule
- Establish project policy and procedures to address the specific needs of the project as a whole, as well as the objectives of each task
- Acquire and apply technical and corporate resources as needed to ensure performance within budget and schedule constraints
- Orient field leaders and support staff concerning the project's special considerations

- Monitor and direct the field leaders
- Develop and meet ongoing project and/or task staffing requirements, including mechanisms to review and evaluate each task product
- Review the work performed on each task to assess its quality, responsiveness, and timeliness
- Review and analyze overall task performance with respect to planned requirements and authorizations
- Approve all external reports (deliverables) before their submission to the U.S. EPA/IEPA
- Ultimately be responsible for the preparation and quality of interim and final reports
- Represent the project team at meetings and public hearings

2.1.6 Warzyn Predesign Investigation Leader

The Warzyn predesign investigation leader (PDIL) for the groundwater phase of the project is Mr. Alan Schmidt. He is responsible for the predesign investigation phase of the Groundwater RD and for leading and coordinating the day-to-day activities of the various resource specialists under his supervision. Specific responsibilities include:

- Provision of day-to-day coordination with the Project Managers on technical issues in specific areas of expertise
- Development and implementation of field-related work plans, assurance of schedule compliance, and adherence to management-developed study requirements
- Coordination and management of field activities including sampling, drilling, and field staff
- Implementation of QC for technical data provided by the field staff including field measurement data
- Adherence to work schedules provided by the Project Managers

- Review and approval of text and graphics required for field team efforts
- Coordination and oversight of technical efforts of subcontractors assisting the field team
- Identification of problems at the field team level, discussion of resolutions with the Project Managers, and provision of communication between team and upper management
- Participation in the preparation of the final report

2.1.7 Technical Staff

The technical staff for this project will be drawn from Warzyn's pool of corporate resources. The technical team staff will be utilized to gather and analyze data, and to prepare various task reports and support materials. All of the designated technical team staff are experienced professionals who possess the degree of specialization and technical competence required to effectively and efficiently perform the required work.

2.1.8 Warzyn Quality Assurance Officer

The Warzyn Quality Assurance Officer (QAO) is Mr. Dan Hall. The QAO will remain independent of direct job involvement and day-to-day operations, and has direct access to corporate executive staff as necessary to resolve any QA dispute. He is responsible for auditing the implementation of the QA program in conformance with the demands of specific investigations, Warzyn's policies, and U.S. EPA requirements. Specific functions and duties include:

- Provide QA audit on various phases of the field operations
- Review and approval of QA plans and procedures
- Provide QA technical assistance to project staff

2.1.9 Laboratory Quality Assurance Officers (Warzyn and IEA)

Duties for the quality assurance officers for Warzyn and IEA include the following:

- Overview laboratory quality assurance
- Overview QA/QC documentation
- Conduct detailed data review
- Decides laboratory corrective action, if required
- Technical representation of laboratory QA procedures
- Preparation of laboratory Standard Operating Procedures
- Approval of QAPjP

2.1.10 Laboratory Operations Manager (Warzyn and IEA)

Duties for Warzyn's and IEA laboratory operations manager include the following:

- Coordinates laboratory analysis
- Supervises in-house chain-of-custody
- Schedules sample analyses
- Oversees data review
- Oversees preparation of analytical reports
- Approves final analytical reports prior to submission to one client

2.2 SPECIALIZED RESPONSIBILITIES

2.2.1 Monitoring and Sampling Operations and QC

- Principal Engineering Firm - Warzyn, Addison, Illinois
- Drilling - To be determined through bidding process
- Field Sampling and Monitoring - Warzyn, Addison, Illinois
- Quality Control - QAO, Warzyn, Madison, Wisconsin

2.2.2 Laboratory Analysis

- Analysis for TCL VOCs and SVOCs by SOW OLMO1.8 as specified in

Table 1-1 will be provided by:

Industrial Environmental Analytical (IEA)
3000 Weston Parkway
Cary, North Carolina 27513
(800) 444-9919
or any other approved laboratory.

- Analysis for purgeable GC VOCs for screening purposes by method 8010/8020 as specified in Table 1-1 will be provided by:

  Warzyn Inc.
  One Science Court
  University Research Park
  Madison, Wisconsin 53711
  (608) 231-4755
  or any other approved laboratory.

- Analysis of samples for selected TAL metals and cyanide by SOW ILMO3.0 (or most current) and indicator parameters as specified in Table 1-1 will be provided by:

  Warzyn Inc.
  One Science Court
  University Research Park
  Madison, Wisconsin, 53711
  (608) 231-4755
  or any other approved laboratory.

The laboratory analysis for parameters to be measured in other phases of the project will be conducted by laboratories listed in the component RD WPs or RD submittals.

2.2.3 Laboratory Data and QC

2.2.3.1 Laboratory Data

- Analytical protocol specified - corresponding laboratory

- Review of analytical protocol - corresponding laboratory technical staff

- Review of analytical procedures - U.S. EPA, Region V, Quality Assurance Section (QAS), Chicago, Illinois
- Internal QA/QC - corresponding laboratory QAO
- Final data review and validation - Chemist, Warzyn, Madison, Wisconsin
- Review of tentatively identified compounds (TICs) and assessment of need for confirmation - Chemist, Warzyn, Madison, Wisconsin

2.2.4 Performance and Systems Audits

2.2.4.1 Field Operations -

- Internal Audits - QAO, Warzyn, Addison, Illinois
- External Audits - will be conducted by U.S. EPA, Region V CRL

2.2.4.2 Analytical Laboratories -

- Internal Audits - corresponding laboratory QAO,
- External Audits - will be conducted by U.S. EPA, Region V, CRL

2.2.4.3 Final Evidence File -

- Final Evidence File Audits - QAO, Warzyn, Addison, Illinois

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QUALITY ASSURANCE OBJECTIVES FOR MEASUREMENT DATA

The overall QA objective is to develop and implement procedures for field sampling, chain-of-custody, laboratory analysis, and reporting that will provide results which are legally defensible in a court of law. Specific procedure for sampling, chain-of-custody, laboratory instruments calibration, laboratory analysis, reporting of data, internal quality control, audits, preventative maintenance of field equipment, and corrective action are described in other sections of this QAPjP. The purpose of this section is to address the specific objectives for accuracy, precision, completeness, representativeness, and comparability.

3.1 LEVEL OF QUALITY CONTROL EFFORT

Field blank, trip blank, duplicate, and matrix spike samples will be analyzed to assess the quality of the data resulting from field sampling. Field and trip blanks consisting of deionized water, will be submitted to the analytical laboratories to provide the means to assess the quality of the data resulting from the field sampling program. Field blank samples are analyzed to check for procedural contamination at the site which may cause sample contamination. Duplicate samples are analyzed to check for sampling and analytical reproducibility. Matrix spikes are analyzed to check for sampling and analytical reproducibility. Matrix spikes provide information about the effect of the sample matrix on the digestion and measurement methodology. Matrix spikes are performed in duplicate for organic analyses, and are hereinafter referred to as matrix spike/matrix spike duplicate (MS/MSD) samples. MS/MSD samples are designated/collected for organic analyses only.

The general level of the QC effort will be one field duplicate and one field blank for every ten or fewer investigative samples. One volatile organic compound

(VOC) trip blank consisting of deionized, ultra pure water, will be included along with each shipment of aqueous VOC samples.

MS/MSD samples are not investigative samples. Aqueous MS/MSD samples must be collected at triple the volume for VOCs and double the volume for extractable organics. One MS/MSD sample will be collected/designated for every 20 or fewer investigative samples per sample matrix (i.e., groundwater, soil). The number of field duplicate and field blank samples to be collected for this site are listed in Table 1-1. A revised table will be submitted as each activity occurs in a QAPjP amendment.

The level of laboratory QC effort for all parameters is summarized in Table 3-5.

3.2 ACCURACY, PRECISION, AND SENSITIVITY OF ANALYSES

The fundamental QA objective with respect to accuracy, precision, and sensitivity of laboratory analytical data is to achieve the QC acceptance criteria of the analytical protocols.

3.2.1. TCL Volatiles and Semi-Volatiles /TAL Inorganics

Laboratory analysis of TCL VOCs and SVOCs and TAL selected metals and cyanide will follow the CLP SOWs (OLMO1.8 [or most current] for TCL organics, ILMO3.0 [or most current] for TAL inorganics). The accuracy, precision, and sensitivity requirements are summarized within the CLP SOWs. Refer to Table 3-1 for the TCL VOCs and SVOCs and the Contract Required Quantitation Limits (CRQLs), and to Table 3-2 for the TAL selected metals and cyanide and the Contract Required Detection Limits (CRDLs).

3.2.2 Purgeable VOCs by GC

The SOP for the Purgeable VOCs is provided in Appendix B1 and will follow GC Volatiles Method 8010/8020. The accuracy and precision requirements of these analyses are summarized in Table 3-5. The sensitivity requirements of these analyses are summarized in Table 3-1.

3.2.3 Indicators

SOPs for the indicator parameters are provided in Appendix B. The accuracy and precision requirements of these analyses are summarized in Table 3-5. The sensitivity requirements of these analyses are summarized in Table 3-3.

3.2.4 Field Measurements

SOPs for the field measurements are provided in Appendix C. The accuracy and precision requirements of these analyses are summarized in Table 3-5. The sensitivity requirements of these analyses are summarized in Table 3-3.

3.2.5 Physical Characteristics (Grain Size)

Determination of physical characteristics will follow the methods referenced in Table 3-4. The accuracy and precision requirements and sensitivity requirements for physical characteristics are summarized in Tables 3-5 and 3-3.

3.3 COMPLETENESS, REPRESENTATIVENESS, AND COMPARABILITY

Completeness is a measure of the amount of valid data obtained from a measurement system compared to the amount that was expected to be obtained under normal conditions. It is expected that the participating laboratories will provide data meeting QC acceptance criteria for 95 percent or more for all samples tested using the SOPs referenced in Section 3.2. Following completion of the analytical testing, the percent completeness will be calculated by the following equation:

$$\text{Completeness} = \frac{\text{Valid Data Obtained}}{\text{Total Data Planned}} \times 100\%$$

Representativeness expresses the degree to which data accurately and precisely represent a characteristic of a population, parameter variations at a sampling point, a process condition, or an environmental condition. Representativeness is a qualitative parameter which is dependent upon the proper design of the sampling program and proper laboratory protocol. The sampling network was designed to provide data representative of site conditions. During development of this network, consideration was given to past site activities, existing analytical data, physical setting and processes, and constraints inherent to the Superfund program. The rationale of the sampling network will be discussed in detail in the SP of the component RD Work Plan.

Representativeness will be satisfied by insuring that the SPs are followed, proper sampling techniques are used, proper analytical procedures are followed, and

holding times of the samples are not exceeded in the laboratory. Representativeness will be assessed by the analysis of field duplicate samples.

Comparability expresses the confidence with which one data set can be compared with another. The extent to which existing and planned analytical data will be comparable, depends on the similarity of sampling and analytical methods. The procedures used to obtain the planned analytical data, as documented in the QAPjP, are expected to provide comparable data. These new analytical data, however, may not be directly comparable to existing data because of differences in procedures and QA objectives.

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4

4

SAMPLING PROCEDURES

4.1 SAMPLING PROCEDURES

A general SP is included as Appendix A to provide standard procedures for anticipated sampling activities. A detailed SP will be developed for each stage of work in the corresponding component RD. The general procedures outlined in Appendix A will be used for Site SPs.

4.2 BOTTLE REQUIREMENTS

The contaminant-free sample containers (bottles) used for analyzing TCL, VOCs and SVOCs, Purgeable GC VOCs, TAL selected metals and cyanide, and indicators parameters will be prepared according to the procedures specified in U.S. EPA's Specifications and Guidance for Obtaining Contaminant-Free Sample Containers, April 1990. It will assure that the bottles used for the sampling activity do not contain contaminants exceeding the level specified in the above-mentioned document. Specifications for the bottles will be verified by checking the supplier's certified statement and analytical results for each bottle lot, and will be documented on a continuing basis. This data will be maintained in the project evidence file, and will be available, if required, for EPA review.

In addition, the data for field blanks and trip blanks will be monitored for contamination, and corrective actions will be taken as soon as a problem is identified. This will be accomplished either by discontinuing the use of a specific bottle lot, contacting the bottle supplier(s) for retesting a representative bottle from a suspect lot, resampling the suspected samples, validating the data taking into account that the contaminants could be introduced by the laboratory (i.e., common lab solvents, sample handling artifacts, etc.) or could be a bottle QC problem, so as to make an educated determination of whether the bottles and hence the data are still usable, etc., whichever is appropriate.

5

5

SAMPLE CUSTODY AND DOCUMENTATION

It is U.S. EPA policy to follow the U.S. EPA sample custody, or chain-of-custody protocols described in "NEIC Policies and Procedures", EPA-330/9-78-DDI-R, Revised June 1985. This custody is in three parts: sample collection, laboratory analysis, and final evidence files. Final evidence files, including all originals of laboratory reports and purge files, are maintained under document control in a secure area.

A sample or evidence file is under your custody if they:

- Are in your possession
- Are in your view, after being in your possession
- Are in your possession and you place them in a secured location
- Are in a designated secure area

5.1 FIELD-SPECIFIC CUSTODY PROCEDURES

The sample packaging and shipment procedures summarized below will insure that the samples will arrive at the laboratory with the chain-of-custody intact.

5.1.1 Initiation of Chain-of-Custody Field Procedures

The field sampler is personally responsible for the care and custody of the samples until they are transferred or properly dispatched to the laboratory. As FEW people as possible should handle the samples.

All bottles will be tagged with sample numbers and locations.

Sample tags are to be completed for each sample using waterproof ink unless prohibited by weather conditions. For example, a logbook notation would explain

that a pencil was used to fill out the sample tag because the ballpoint pen would not function in freezing weather.

5.1.2 Field Logbooks/Documentation

Field logbooks will provide the means of recording data collecting activities performed. As such, entries will be described in as much detail as possible so that persons going to the site could re-construct a particular situation without reliance on memory.

Field logbooks will be bound, field survey books or notebooks. Logbooks will be assigned to field personnel, but will be stored in the document control center when not in use. Each logbook will be identified by the project-specific document number.

The title page of each logbook will contain the following:

- Person to whom the logbook is assigned
- Logbook number
- Project name
- Project start date
- Project end date

Entries into the logbook will contain a variety of information. At the beginning of each entry, the date, start time, weather, names of all sampling team members present, level of personal protection being used, and the signature of the person making the entry will be entered. The names of visitors to the site, field sampling or investigation team personnel, and the purpose of their visit will also be recorded in the field logbook.

Measurements made and samples collected will be recorded. All entries will be made in ink, and no erasures will be made. If an incorrect entry is made, the information will be crossed out with a single strike mark. Whenever a sample is collected, or a measurement is made, a detailed description of the location of the station, which includes compass and distance measurements, shall be recorded. The number of the photographs taken of the station, if any, will also be noted. The equipment used to make measurements will be identified, along with the date of calibration.

Samples will be collected following the sampling procedures documented in the SP developed for that sampling activity. The equipment used to collect samples

will be noted, along with the time of sampling, sample description, depth at which the sample was collected, volume and number of containers. A sample identification number will be assigned prior to sample collection. Field duplicate samples, which will receive an entirely separate sample identification number, will be noted under sample description.

5.1.3 Transfer of Custody and Shipment Procedures

Samples are accompanied by a properly completed chain-of-custody form. The sample numbers and locations will be listed on the chain-of-custody form. When transferring the possession of samples, the individuals relinquishing and receiving will sign, date, and note the time on the record. This record documents transfer of custody of the samples from the sampler to another person, to a mobile laboratory, to the permanent laboratory, or to/from a secure storage area.

Samples will be properly packaged for shipment and dispatched to the appropriate laboratory for analysis, with a separate signed custody record enclosed in each sample box or cooler. Shipping containers will be locked and secured with strapping tape and custody seals for shipment to the laboratory. The preferred procedure includes the use of a custody seal attached to the front right and back left of the cooler. The custody seals are covered with clear plastic tape. The cooler is strapped shut with strapping tape in a least two locations.

Whenever samples are split with another source or government agency, a separate sample custody record is prepared for those samples, and marked to indicate with whom the samples are being split. The person relinquishing the samples to the facility or agency should request the representative's signature acknowledging the sample receipt. If the representative is unavailable or refuses, the person relinquishing the samples should note this in the "received by" space of the custody form.

All shipments will be accompanied by the chain-of-custody record identifying the contents. The original record will accompany the shipment, and the pink and yellow copies will be retained by the sampler for returning to the sampling office.

If the samples are sent by common carrier, a bill of lading should be used. Receipts of bills of lading will be retained as part of the permanent documentation. If sent by mail, the package will be registered with return receipt requested. Commercial carriers are not required to sign off on the custody forms as long as the custody forms are sealed inside the sample cooler, and the custody

seals remain intact. The person shipping the samples should note the carrier name and airbill number on the chain-of-custody record.

5.2 LABORATORY CHAIN-OF-CUSTODY PROCEDURES

Laboratory custody procedures for the sample receiving and log-in; sample storage; tracking during sample preparation and analysis; and storage of data are described in the individual laboratory custody SOPs provided in Appendix D.

5.3 FINAL EVIDENCE FILE CUSTODY PROCEDURE

The final evidence file for the WRL Site will be located at and maintained by Warzyn. The content of the evidence file will include all relevant records, reports, correspondence, logs, field logbooks, laboratory sample preparation and analysis logbooks/documentation, analytical data packages, pictures, subcontractor reports, chain-of-custody records/forms, data review reports, etc.. The evidence file will be under custody of the file custodian in a locked, secured area.

6

6

CALIBRATION PROCEDURES AND FREQUENCY

This section describes procedures for maintaining the accuracy of all the instruments and measuring equipment which are used for conducting field tests and laboratory analyses. These instruments and equipment should be calibrated prior to each use or on a scheduled, periodic basis.

6.1 FIELD INSTRUMENTS/EQUIPMENT

Instruments and equipment used to gather, generate, or measure environmental data will be calibrated with sufficient frequency and in such a manner that accuracy and reproducibility of results are consistent with the manufacturer's specifications.

Equipment used during the field sampling will be examined to certify that it is in operating condition. This includes checking the manufacturer's operating manual and the instructions for each instrument to determine that all prior equipment problems are not overlooked, and all necessary repairs to equipment have been carried out.

Calibration procedures and frequencies for field instruments are governed by the specific field SOPs provided in Appendix C.

6.2 LABORATORY INSTRUMENTS

Procedures for the calibration of laboratory instruments must be established and maintained so that equipment is functioning properly and that data collected are accurate and reliable. Requirements include step-by-step calibration procedures, frequency of re-calibration, equipment maintenance logs, instrument accuracy criteria, corrective action procedures and equipment limitations (e.g., working ranges), and are described, in detail, in the SOPs provided in Appendix B.

7

ANALYTICAL PROCEDURES

Samples collected during field sampling activities will be analyzed by Warzyn, IEA or another approved laboratory. Refer to Table 1-1 for matrices, number of samples, parameters, and the laboratory performing the analysis. Sampling, analysis, and designated laboratories for additional work will be described in the component RD documents.

7.1 FIELD SCREENING ANALYTICAL PROTOCOLS

The procedures for the field measurements are described in the SOPs provided in Appendix C.

7.2 LABORATORY ANALYSES

Samples for TAL selected metals and cyanide and analyzed by Warzyn will follow the SOW ILMO3.0 (or most current).

Samples for TCL VOCs and SVOCs analyzed by IEA or another approved laboratory will follow the SOW OLMO1.8 (or most current).

Samples for indicator parameters analyzed by Warzyn will follow the SOPs in Appendix B.

Samples for Purgeable GC VOCs for screening purposes analyzed by Warzyn will follow the SOP in Appendix B for GC Volatiles Method 8010/8020.

Refer to Table 7-1 for a summary of analytical methods for analyses.

8

8

INTERNAL QUALITY CONTROL CHECK

8.1 FIELD SAMPLE COLLECTION

The assessment of field sampling precision and accuracy will be made through the collection of field duplicates and field blanks in accordance with the procedures described in the field SPs. Refer to Table 1-1 for a summary of sample numbers and required field QC samples for anticipated sampling activities.

8.2 FIELD MEASUREMENTS

QC procedures for field measurements are limited where applicable to checking the reproducibility of the measurements by obtaining multiple readings on a single sample or standard, and by calibrating the instruments. Refer to Table 3-5 for a summary of QC requirements.

8.3 LABORATORY ANALYSIS

The laboratories used for the analysis of samples for the RD/RA phase have written QA/QC programs which provide rules and guidelines to ensure the reliability and validity of work conducted at the laboratory. Compliance with the QA/QC program is coordinated and monitored by the laboratory QAOs, which are independent of the operating departments. Laboratory procedures used are documented in writing and are provided or referenced in this QAPjP. Internal quality control checks are an integral part of the analytical methods, and are discussed in detail within the analytical procedures. The overall objectives of the internal quality control checks are to verify the established precision, accuracy and integrity of the methodology and to support the technical validity of the data. Where appropriate, internal quality control checks will include method blanks, preparation/reagent blanks, calibration check samples, laboratory duplicates, matrix spikes and continuing calibration standards.

The required quality control frequency and performance criteria for the indicator parameters are summarized in Table 3-5.

The performing laboratories will document, in each data package provided, that both initial and ongoing instrument and analytical QC functions have been met. Samples analyzed in non-conformance with the QC criteria set forth, will be reanalyzed by the laboratory. It is expected that sufficient sample volume will be collected for reanalysis.

9

9

DATA REDUCTION, VALIDATION, AND REPORTING

9.1 FIELD MEASUREMENTS AND SAMPLE COLLECTION

Raw data from field measurements and sample collection activities will be appropriately recorded in the field logbook. Data will be reviewed to ensure procedures were followed and QC requirements were met, however, no formal data validation effort will be performed. If the data are to be used in the project reports, they will be reduced onto data summary tables.

9.2 LABORATORY SERVICES

9.2.1 Data Reduction

Each laboratory is responsible for identification, quantification, data reporting, and data deliverables for the analyses performed. Data reduction of parameters will follow the requirements set forth in the corresponding SOP. Deliverables include full data packages containing raw data, summaries of calibration standards, duplicates, spikes, blanks, and performance evaluation samples.

Full data packages will not be developed for the Purgeable GC VOC data since these samples will be used primarily to locate the screened interval in the wells and will require a 24-hr turnaround.

9.2.2 Data Validation

Analytical data will be used to determine extent and delineation of contamination for engineering purposes. This data has a DQO of Level 4 and will require full data validation.

The data generated for purgeable VOC analysis while drilling is intended as field screening for the zone of highest VOC contamination in groundwater for well screen placement purposes. Thus, it will not require data validation.

Refer to Table 1-3 for a summary of data generating activities, intended data uses, and associated DQOs for the site. The table may be modified in a QAPjP amendment based on component RD requirements.

9.2.3 Data Reporting

Analytical data generated for the site will be computerized in a format organized to facilitate data review and evaluation. The computerized data set will include the data qualifiers provided by the performing laboratory in accordance with the SOPs, as well as qualifiers added by the data reviewer in accordance with the SOPs.

The laboratory-provided qualifiers will include such items as:

- Non-detects
- Concentration below required detection limit
- Estimated concentration due to poor QC data
- Concentration of chemical also found in the laboratory blank

A summary of the analytical data will be incorporated into the corresponding report.

10

PERFORMANCE AND SYSTEM AUDITS

Performance and system audits of both field and laboratory activities will be conducted to determine that sampling and analysis are performed in accordance with the procedures established in the QAPjP and the corresponding component RDs. The audits of field and laboratory activities include two separate independent parts: internal and external audits.

10.1 FIELD AUDITS

10.1.1 Internal Audits

Internal audits of field activities (sampling and measurements) will be conducted by the Warzyn QAO. The purpose of the field audit will be to evaluate and document adherence to procedures described in the QAPjP and the corresponding QAPjP addendum. The audit will include review of field activities, sample documentation, chain-of-custody forms, field logbooks, and sampling and decontamination activities. Follow-up audits will be conducted to correct deficiencies, and to document that QA procedures are maintained throughout the investigation.

10.1.2 External Audits

External audits will be the responsibility of the U.S. EPA, Region V, RPM.

10.2 LABORATORY AUDITS

10.2.1 Internal Audits

The purpose of the internal laboratory audit is to evaluate and document adherence to analytical procedures described in this QAPjP. Internal audits of the participating laboratories are the responsibility of the individual laboratory QAO. System audits will include examination of laboratory documentation on sample receiving, sample log-in, sample storage, chain-of-custody procedure, sample preparation and analysis, instrument operating records, etc., and will be performed annually. Performance audits consisting of blind QC samples prepared and submitted to the laboratory for analysis, will be performed quarterly. Results of these blind QC samples will be reviewed by the laboratory QAO.

10.2.2 External Audits

The U.S. EPA, Region V, Central Regional Laboratory (CRL) may audit performing laboratories and provide recommendations for approval of the laboratory for the requested analyses to the U.S. EPA RPM. The audit may consist of a review of analytical and chain-of-custody procedures, evaluation of performance samples, and may also include an on-site audit of each participating laboratory.

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11

PREVENTATIVE MAINTENANCE

11.1 FIELD INSTRUMENTS/EQUIPMENT

Field equipment used for the project may include: thermometers, pH meter, conductivity meter, gas-tech meter, electronic water level indicator, and sampling and filtering equipment. Specific preventative maintenance procedures to be followed for field equipment are those recommended by the manufacturer.

Field instruments will be checked and calibrated before they are transported to the field. These instruments will be checked and calibrated daily before use. Calibration checks will be performed as noted in Table 3-5.

Critical spare parts such as electrodes, batteries, and pH probes will be kept on-site to minimize instrument down time. Backup instruments and equipment will be available to avoid delays in the field schedule.

Routine preventative maintenance schedules are summarized in Table 11-1.

11.2 LABORATORY INSTRUMENTS

Preventative maintenance of laboratory instruments will be as directed with manufacturer's specifications, instrument operating procedures, and analytical methods. Maintenance is carried out on a regular, scheduled basis, and is documented in the instrument service logbook for each instrument. Emergency repair or scheduled manufacturer's maintenance is provided by an on-site technician or maintenance contract with the factory representatives.

Routine preventative maintenance schedules are summarized in Table 11-1.

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

12.1 FIELD MEASUREMENTS

Field data will be assessed by the site QAO. The site QAO will review the field results for compliance with the established QC criteria that are specified in the QAPjP and the corresponding QAPjP addendum. Accuracy of field measurements will be assessed using daily instrument calibration, calibration check, and blank data. Precision will be assessed on the basis of reproducibility by taking multiple readings of a single sample. Data completeness will be calculated as follows:

$$\text{Completeness} = \frac{\text{Valid Data Obtained}}{\text{Total Data Planned}} \times 100\%$$

12.2 LABORATORY DATA

12.2.1 Precision

Precision of laboratory analysis will be assessed by comparing the analytical results between MS/MSD for organic analysis, and laboratory duplicate analyses for inorganic and indicator analysis. The relative percent difference (RPD) will be calculated for each pair of duplicate analysis results as follows:

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

where, S = first sample value (original or MS value)
D = Second sample value (duplicate or MSD value)

12.2.2 Accuracy

Accuracy of laboratory results will be assessed based on the established QC criteria that are described in Section 3 of this QAPjP, using the analytical results of method blanks, matrix spike samples, field blanks, and bottle blanks. The percent recovery (%R) will be calculated as follows:

$$\%R = \frac{A - B}{C} \times 100\%$$

where, A = The analyte concentration determined experimentally from the spiked sample

B = The background level determined by a separate analysis of the unspiked sample

C = The amount of the spike added

12.2.3 Completeness

The data completeness of laboratory analysis results will be assessed for compliance with the amount of data required for decision making. Completeness is calculated by the following formula:

$$\text{Completeness} = \frac{\text{Valid Data Obtained}}{\text{Total Data Planned}} \times 100\%$$

12.2.4 Sensitivity

The achievement of method detection limits depend on instrument sensitivity and matrix effects. It is important to monitor the instrument sensitivity to ensure the data quality through constant instrument performance. The instrument sensitivity will be monitored through the analysis of method blanks, calibration check samples, and laboratory control samples.

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CORRECTIVE ACTION

Corrective actions may be required for two classes of problems: analytical and equipment problems, and noncompliance problems. Analytical and equipment problems may occur during sampling and sample handling, sample preparation, laboratory instrumental analysis, and data review.

For noncompliance problems, a formal corrective action program will be determined and implemented at the time the problem is identified. The person who identifies the problem is responsible for notifying the Warzyn Project Manager, who in turn will notify the EPA RPM. If the problem is analytical in nature, information on these problems will be promptly communicated to the Warzyn Project Manager, who in turn will notify the RPM or EPA QAS. Implementation of corrective action will be confirmed in writing through the same channels.

Any noncompliance with the established quality control procedures in the QAPjP or the corresponding QAPjP addendum will be identified and corrected in accordance with the QAPjP. The EPA RPM, or his designee will issue a non-conformance report for each non-conformance condition.

Corrective actions will be implemented and documented in the field record book. No staff member will initiate corrective action without prior communication of findings through the proper channels. If corrective actions are insufficient, work may be stopped by stop-work order by the U.S. EPA RPM.

13.1 SAMPLE COLLECTION/FIELD MEASUREMENT

Technical staff and project personnel will be responsible for reporting all suspected technical or QA non-conformances or suspected deficiencies of any activity or issued document by reporting the situation to the Warzyn QAO or his designee. This manager, in consultation with the Warzyn Project Manager, will be responsible for assessing the suspected problems and making a decision based

on the potential for the situation to impact the quality of the data. If it is determined that the situation warrants a reportable non-conformance requiring corrective action, then a non-conformance report will be initiated by the manager.

The manager will be responsible for ensuring that corrective action for non-conformances are initiated by:

- Evaluating all reported non-conformances
- Controlling additional work on non-conforming items
- Determining disposition or action to be taken
- Maintaining a log of non-conformances
- Reviewing non-conformance reports and corrective actions taken
- Ensuring non-conformance reports are included in the final site documentation in the project files

If appropriate, the Warzyn Project Manager will determine that, no additional work that is dependent on the non-conforming activity, is performed until the corrective actions are completed.

Corrective action for field measurements may include:

- Repeat the measurement to check the error
- Check for all proper adjustments for ambient conditions such as temperature
- Check the batteries
- Re-calibration
- Check the calibration
- Replace the instrument or measurement devices
- Stop work (if necessary)

The Warzyn Predesign Investigation Leader, or his designee, is responsible for all site activities. In this role, the Warzyn PDIL, at times is required to adjust the site programs to accommodate the site-specific needs. When it becomes necessary to modify a program, the Warzyn PDIL notifies the Warzyn Project Manager of the anticipated change and implements the necessary changes after obtaining the approval of the Project Manager. The change in the program will be documented and signed by the appropriate personnel.

The Warzyn PDIL Leader for the site is responsible for the controlling, tracking, and implementation of the identified changes. Reports on all changes will be distributed to all affected parties, including the U.S. EPA RPM, the Warzyn Project Manager, and the WRS Project Coordinator.

13.2 LABORATORY ANALYSIS

Corrective actions are required when an out-of-control event or potential out-of-control event is noted. The investigative action taken is dependent on the analysis and the event. Laboratory personnel are alerted that corrective actions may be necessary if:

- QC data are outside the warning or acceptable windows for precision and accuracy
- Blanks contain target analytes above acceptable levels
- Undesirable trends are detected in spike recoveries or RPD between duplicates
- There are unusual changes in detection limits
- Deficiencies are detected by the QA Department during internal or external audits , or from the results of performance evaluation samples
- Inquiries concerning data quality are received.

Corrective action procedures are often handled at the bench level, by the analyst who reviews the sample preparation procedure for possible errors and checks the instrument calibration, spike and calibration mixes, instrument sensitivity, etc. If

the problem persists, or cannot be identified, the matter is referred to the laboratory supervisor, manager, or QAO for further investigation. Once resolved, full documentation of the corrective action procedure is filed with the QA Department.

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QUALITY ASSURANCE REPORTS TO MANAGEMENT

Reports will be submitted to the U.S. EPA as described in the RD/RA Work Plan and component RD Work Plans for the WRL Site. Analytical data will be submitted as a part of the appropriate reports.

TABLES

TABLE 1-1

Sample Type and Estimated Sample Numbers
Winnebago Reclamation Landfill Site RD/RA

Sample[1] Matrix	Lab[2]	No. of Samples	Field Duplicates	Field[3] Blanks	MS/MSD[4]	Total No. Samples	Test[5,6] Parameters	Field Parameters
Groundwater Extraction and Treatment								
Groundwater Phase I	IEA	19	2	2	1	23	TCL VOCs, SVOCs TAL Selected Metals and Cyanide Indicators	pH, Conductivity Temperature
	Warzyn	19	2	2	1	23		
Groundwater Phase II Sampling while drilling	Warzyn	6			--	6	Purgeable GC VOCs	--
Geotechnical Sampling	Warzyn	4	--	--	--	4	Grain size	--
Monitoring well sampling	IEA	9	1	1	1	11	TCL VOCs, SVOCs TAL Selected Metals and Cyanide Indicators	pH, Conductivity Temperature
	Warzyn	9	1	1	1	11		
Groundwater Phase III	IEA	8	1	1	1	9	TCL VOCs, SVOCs TAL Selected Metals and Cyanide Indicators	pH, Conductivity Temperature
	Warzyn	8	1	1	1	9		
Gas/Leachate Extraction and Treatment								
Activity	TBD	TBD	TBD	TBD	TBD	TBD	TBD	TBD
Final Cover System								
Activity	TBD	TBD	TBD	TBD	TBD	TBD	TBD	TBD

TABLE 1-1

Footnotes:

(1) Samples will be considered low concentration, and will be packaged and shipped accordingly.

(2) Warzyn Analytical Laboratory
One Science Court
University Research Park
Madison, WI 53711

Industrial Environmental
Analytical (IEA)
3000 Weston Parkway
Cary, NC 27513

TBD = To be determined in a future component WP.

(3) A trip blank for VOC analysis will be included with each cooler shipped for aqueous groundwater samples. Trip blanks are not included in the total number of samples.

(4) Extra Volume Requirement: Extra volume is required for the MS/MSD quality control requirement (triple volume for VOCs double the volume for SVOCs). Inorganics and general water quality indicator parameters require duplicate and spike analyses, however, do not require additional sample volume to meet the specified QC. MS/MSD samples are not included in the total number of samples.

(5) Refer to Tables 3-1, 3-2, and 3-3 for the TCL VOCs and SVOCs, Purgeable GC VOCs, TAL Selected Metals and Cyanide, and Indicators parameter lists and required detection limits, respectively.

(6) Groundwater samples for metals and TDS analysis will be field filtered through a 0.45 micron filter prior to the addition of preservatives (if any).

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TABLE 1-2

Sample Quantities, Containers, Preservatives and Packaging Requirements
Winnebago Reclamation Landfill Site RD/RA

Analysis[1]	Bottles and Jars	Preservation	Technical Holding Time[2]	Volume of Samples	Shipping	Normal Packaging[3]
GROUNDWATER EXTRACTION AND TREATMENT						
Low Concentration (Organics)						
Groundwater						
TCL Volatiles and Purgeable GC VOCs	Four 40-mL volatile organic analysis (VOA) vial.	1:1 HCL (2 drops/vial) Iced to 4°C.	14 days from sampling date	Fill completely no headspace	Shipped daily by overnight carrier	Vermiculite
TCL SVOCs	Two 1-liter amber glass	Iced to 4°C	5 days until *extraction, 40* days from sampling date	Fill to shoulder *of bottle*	Shipped daily	Vermiculite
Low Concentration (Inorganics)						
Groundwater						
TAL Metals	One 1-liter high density polyethylene bottle	Field filter through 0.45 um filter. HNO_3 to pH<2. Iced to 4°C	180 days from sampling (26 days from sampling for mercury)	Fill to shoulder of bottle	Shipped daily by overnight carrier	Vermiculite
Cyanide	One 1-liter high density polyethylene bottle	NaOH to pH>12 Iced to 4°C	14 days from sampling date	Fill to shoulder of bottle	Shipped daily by overnight carrier	Vermiculite
Water Quality Indicator Parameters						
Groundwater						
TOC, COD, Ammonia-N Nitrate + Nitrite	One 1-liter density polyethylene bottle	H_2SO_4 to pH<2. Iced to 4°C.	28 days	Fill to shoulder of bottle	Shipped daily by overnight carrier	Vermiculite
BOD_5	One 1-liter high density polyethylene bottle	Iced to 4°C	48 hours	Fill to shoulder of bottle	Shipped daily by overnight carrier	Vermiculite

TABLE 1-2

Analysis[1]	Bottles and Jars	Preservation	Technical Holding Time[2]	Volume of Samples	Shipping	Normal Packaging[3]
Alkalinity, Chloride, Sulfate, and TSS	One 1-liter high density polyethylene bottle	Iced to 4°C	28 days (14 days alkalinity)	Fill to shoulder of bottle	Shipped daily by overnight carrier	Vermiculite
TDS	One 500-mL high density polyethylene bottle	Field filter through 0.45 um Iced to 4°C	7 days	Fill to shoulder of bottle	Shipped daily by overnight carrier	Vermiculite

GAS LEACHATE EXTRACTION AND TREATMENT

TBD

FINAL COVER SYSTEM

Physical Analysis

TBD

Footnotes:

(1) The analysis for future phases will be described in the corresponding component RD WP.
(2) Technical hold times begin on the date sampled.
(3) The packing material should completely cushion the sample bottles - bottom, sides and top.

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[mad-407-105b]
10009721/157

TABLE 1-3

Summary of Data Generating Activities and Associated Quality Objectives
Winnebago Reclamation Landfill Site RD/RA

Activity	Description	Intended Data Usages	Parameters[3]	Data Quality Objective	Anticipated No. of Investigative Samples
Groundwater Extraction and Treatment					
Groundwater Sampling[1]	Sample and analyze groundwater from selected wells in 2 rounds	Extent and delineation of the contamination on site for engineering purposes	TCL VOCs, SVOCs TAL Metals and Cyanide Indicators	Level 4 Level 4 Level 4	19 19 19
	Groundwater sampling collected while drilling	Locate the screened intervals in the wells with 24-hr sample turnaround	Purgeable GC VOCs	Level 2	6
Soil Sampling	Sample and analyze soils while drilling	Physical characteristics of soil	Grain size	Level 3	4
Gas/Leachate Extraction and Treatment					
TBD[2]					
Final Cover System					
TBD					

Footnotes:

(1) Groundwater samples are to be filtered for dissolved TAL Metals analysis and TDS.
(2) TBD = To be determined in a future component RD WP.
(3) Refer to Tables 3-1, 3-2, and 3-3 for the TCL VOCs, TAL Metals, and Indicators parameters lists and required detection limits, respectively.

TABLE 3-1

Target Compound List (TCL), Purgeable GC VOCs, and Contract Required Quantitation Limits (CRQLs)

	Compound	CRQLs TCL[1] Groundwater (ug/L)	CRQLs Purgeable GC VOCs[2] Groundwater (ug/L)
	Volatiles		
1.	Chloromethane	10	2
2.	Bromomethane	10	1
3.	Vinyl chloride	10	1
4.	Chloroethane	10	2
5.	Methylene chloride	10	3
6.	Acetone	10	--
7.	Carbon disulfide	10	--
8.	1,1-Dichloroethene	10	1
9.	1,1-Dichloroethane	10	1
10.	1,2-Dichloroethene (total)	10	1
11.	Chloroform	10	1
12.	1,2-Dichloroethane	10	1
13.	2-Butanone (MEK)	10	--
14.	1,1,1-Trichloroethane	10	1
15.	Carbon tetrachloride	10	1
16.	Bromodichloromethane	10	1
17.	1,2-Dichloropropane	10	--
18.	cis-1,3-Dichloropropene	10	--
19.	Trichloroethene	10	1
20.	Dibromochloromethane	10	1
21.	1,1,2-Trichloroethane	10	1
22.	Benzene	10	1
23.	trans-1,3-Dichloropropene	10	--
24.	Bromoform	10	1
25.	4-Methyl-2-pentanone (MIBK)	10	--
26.	2-Hexanone	10	--
27.	Tetrachloroethene	10	1

TABLE 3-1

Compound	CRQLs TCL VOCs(1) Groundwater (ug/L)	CRQLs Purgable GC VOCs(2) Groundwater (ug/L)
Volatiles		
28. Toluene	10	1
29. 1,1,2,2-Tetrachloroethane	10	1
30. Chlorobenzene	10	1
31. Ethylbenzene	10	1
32. Styrene	10	1
33. Xylenes (total)	-	3
34. Cis 1,2-Dichloroethene	-	1
35. trans 1,2-Dichloroethene	-	1
36. 1,2-Dibromoethane	-	1
37. 1,2-Dibromo-3-chloropropane	-	3
38. 1,3-Dichlorobenzene	-	1
39. 1,4-Dichlorobenzene	-	1
40. 1,2-Dichlorobenzene	-	1
41. Bromo chloromethane	-	1
Semi-Volatiles		
1. bis(2ethylhexyl)phthalate	10	-

Footnotes:

(1) TCL VOCs and SVOCs by SOW OLMO1.8 (or most current)
(2) Purgeable VOCs by method found in Appendix B1

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[mad-407-105d]
10009721/157

TABLE 3-2

Target Analyte List (TAL) for Selected Metals and Cyanide in Groundwater

Analyte	Reported[1] Detection Limit (ug/L)
1. Arsenic	10
2. Barium	200
3. Cadmium	5
4. Calcium	5000
5. Cobalt	50
6. Iron	100
7. Magnesium	5000
8. Manganese	15
9. Nickel	40
10. Potassium	5000
11. Sodium	5000
12. Thallium	10
13. Zinc	20
14. Cyanide	10

Footnotes:

(1) SOW ILMO3.0 (or most current)

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[mad-407-105e]
10009721/157

TABLE 3-3

Indicators, Field Measurements, Physical Characteristics, and Non-Aqueous Parameter Lists and Required Detection Limits

Water Quality Indicator Parameter	Required Detection Limit (mg/L)
Alkalinity, Total	10
Chloride	2
Sulfate	10
Ammonia Nitrogen	0.10
Total Dissolved Solids (TDS)	20
Total Suspended Solids (TSS)	2
Total Organic Carbon (TOC)	1.0
Chemical Oxygen Demand (COD)	20
Biological Oxygen Demand (BOD-5 day)	1
Nitrate + Nitrite Nitrogen	0.02

Field Measurement Parameter	Required Detection Limit
Aqueous:	
pH (s.u.)	sensitivity of 0.05
Conductivity @25 Deg. C (umhos/cm)	10
Temperature (Deg. C)	to nearest 0.5 degree
Non-aqueous:	
Methane	1%
Oxygen	1%
Carbon Dioxide	1%

Physical Characteristic Parameter	Required Detection Limit
Grain Size	NA

General Note:

1. NA = Not applicable to method.

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[mad-407-105f]
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TABLE 3-4

Summary of Physical Characteristics Method References

Test	Method Reference[1,2]
Grain Size	ASTM D422

Footnotes:

(1) ASTM = American Society for Testing and Materials
(2)

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[mad-407-105g]
10009721/157

TABLE 3-5

Summary of Quality Control Requirements
Winnebago Reclamation Landfill Site RD/RA

Parameter	Audit	Frequency[1]	Limits[2]
AQUEOUS			
TCL VOCs and SVOCs	Requirements per OLMO1.8 (or most current)		
Purgeable GC Volatiles	Method Blank	Each day before samples are analyzed	<Detection limit (DL)
	Check Standard	After calibration	±30% Recovery
	Field Duplicate	1 per 10 samples	20% RPD (± 2xDL if sample concentration is <5 x DL)
	Field Blank	1 per 10 samples	No Limits
	Matrix Spike/ Matrix Spike Duplicate	1 per 10	70% to 130% Recovery 20% RPD
	Laboratory Fortified Blank	1 per 15	<DL
	Surrogates	Each sample	Laboratory established control limits. 50% to 150% Recovery
	Initial Calibration Criteria	Beginning of analytical run	Correlation coefficient ≥0.995
	Continuing Calibration Verification	Initially and end of analytical run	70%-130% (50%-150% gases)
TAL Metals and Cyanide	Requirements per ILMO3.0 (or most current)		
Alkalinity, Chloride, Sulfate	Lab Blank	After calibration, every 10 samples, and at the end of the run.	< Detection Limit (DL)
	Check Standard	After calibration, every 10 samples, and at the end of the run.	90 - 110 % Recovery
	ICB/CCB	After calibration, every ten samples, and at the end of the run	<DL
	ICV/CCV	After calibration, every ten samples, and at the end of the run	90%-110% recovery

TABLE 3-5

Parameter	Audit	Frequency[1]	Limits[2]
	EPA QC Reference Standard	1 per set	80 - 120 % Recovery
	Lab Duplicate	1 per 10 samples	10% RPD (± 2xDL if sample concentration is <5 x DL)
	Matrix Spike	1 per 10 samples	85 - 115 % Recovery
TDS and TSS	Lab Blank	1 per set	< DL
	EPA QC Reference Standard	1 per set	80 - 120 % Recovery
	Lab Duplicate	1 per 10 samples	10% RPD (± 2xDL if sample concentration is <5 x DL)
Ammonia Nitrogen and Nitrate + Nitrite Nitrogen	Lab Blank	After calibration, every 10 samples, and at the end of the run.	< DL
	Check Standard	After calibration, every 10 samples, and at the end of the run.	90 - 110 % Recovery
	Preparation Blank	1 per set	< DL
	EPA QC Reference Standard	1 per set	80 - 120 % Recovery
	Lab Duplicate	1 per 10 samples	20% RPD (± 2xDL if sample concentration is <5 x DL)
	Matrix Spike	1 per 10 samples	85 - 115 % Recovery
BOD_5	Lab Blank	1 per 10 samples	< DL
	Check Standard (Glucose-Glutamic Acid, 200 mg/L)	1 per 10 samples	200 ± 37 mg/L
	EPA QC Reference Standard	1 per set	80 - 120 % Recovery
	Lab Duplicate	1 per 10 samples	10% RPD (± 2xDL if sample concentration is <5 x DL)

TABLE 3-5

Parameter	Audit	Frequency(1)	Limits(2)
COD	Lab Blank	1 per 10 samples	< DL
	Check Standard	1 per 10 samples	90 - 110 % Recovery
	EPA QC Reference Standard	1 per set	80 - 120 % Recovery
	Lab Duplicate	1 per 10 samples	10% RPD (± 2xDL if sample concentration is <5 x DL)
	Matrix Spike	1 per 10 samples	85 - 115 % Recovery
TOC	Lab Blank	1 per 10 samples	< DL
	Check Standard	1 per 10 samples	90 - 110 % Recovery
	EPA QC Reference Standard	1 per set	80 - 120 % Recovery
	Lab Duplicate	1 per 10 samples	20% RPD (± 2xDL if sample concentration is <5 x DL)
	Matrix Spike	1 per 10 samples	75 - 125 % Recovery
pH (Field)	Check Standard	1 per 10 samples	± 0.05 pH unit of buffer selection
	Duplicate	1 per 10 samples	± 0.2 pH unit
Specific Conductance (Field)	Check Standard	1 per 10 samples	± 10% of standard
	Duplicate	1 per 10 samples	20% RPD (± 2xDL if sample concentration is <5 x DL)
Temperature (as part of Spec. Cond.)	Duplicate	1 per 10 samples	± 0.5 °C
NON-AQUEOUS			
Grain Size Distribution	Lab Duplicate	1 per 10 samples	40% RPD or <2% by weight
CH_4, O_2, CO_2	Duplicate	1 per 10 readings	± 1 %

Footnotes:

(1) Frequencies apply to each individual matrix.

(2) Refer to Appendix B for required detection limits for each analyte.

(3) Quality Control Requirements for future project phases will be included in the corresponding component RD WP.

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[mad-407-105h]
10009721/105h

TABLE 7-1

Summary of Analytical Methods
Winnebago Reclamation Landfill Site RD/RA

Parameter	SOW(1) or SOP(2)	Method(3,4)
TCL VOCs and SVOCs	OLMO1.8	--
Purgeable GC VOCs	Volatiles	SW-846 (8010/8020)
TAL Selected Metals and Cyaide	ILMO3.0	
Aqueous:		
Total Alkalinity	la-200	EPA 310.2
Chloride	la-202	EPA 325.2
Sulfate	la-216	EPA 375.2
Ammonia Nitrogen	wc-321	EPA 350.2
Total Dissolved Solids	wc-336	EPA 160.1
Total Suspended Solids	wc-337	EPA 160.2
Total Organic Carbon	ex-812	EPA 415.1
Chemical Oxygen Demand	wc-305	EPA 410.4
Biological Oxygen Demand-5 day	wc-302	EPA 405.1
Nitrate + Nitrite Nitrogen	la-208	EPA-353.2
Field Measurements:		
pH	FIELDSOP-998	EPA 150.1
Specific conductance	FIELDSOP-999	EPA 120.1
Temperature	FIELDSOP-987	EPA 170.1

Footnotes:

(1) SOW refers to the Contract Laboratory Program Statement of Work.
(2) SOP refers to the Warzyn or IEA laboratory document number.
(3) Method refers to the published analytical reference.
(4) SCA = Soil Chemical Analysis, M.L. Jackson, First Course, 6th Print, 1970.

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[mad-607-105i]
10009721/157

TABLE 11-1

Routine Preventative Maintenance Procedures and Schedules
Winnebago Reclamation Landfill Site RD/RA

Instrument	Maintenance Procedures/Schedule	Spare Parts in Stock
IEA Instrumentation:		
Gas Chromatograph/Gas Spectrometry (GC/MS)-Volatiles	1. Change septa as needed. 2. Column maintenance as needed. 3. Clean injection port liner as needed. 4. Backflush purge and trap device as needed. 5. Change trap as needed.	1. Septa 2. GC columns 3. Syringes 4. Various electronic components 5. Traps
Warzyn Instrumentation:		
Graphite Furnace Atomic Absorption Spectrometer	1. Clean furnace chamber, contact rings, and furnace windows with alcohol daily. 2. Change contact rings as needed. 3. Ensure sufficient supply of D.I. H_20 in rinse bottle, daily. 4. Check gas pressure, daily.	1. Contact rings 2. Graphite tubes 3. Spare shroud
Flame Atomic Absorption Spectrometer	1. Clean burner head when necessary. 2. Check water trap to ensure it is full and clean prior to analysis. 3. Empty drain vessel when necessary. 4. Clean/inspect/optimize nebulizer monthly or as needed. 5. Clean & check o-rings monthly or as needed. 6. Rinse spray chamber with D.I. H_2O when run is completed.	1. Burner Head 2. Nebulizer tubing 3. O-rings 3. O-rings 4. Nebulizer
Inductively Coupled Plasma Spectrometer (ICP)	A. Daily 1. Check peristaltic pump rollers to ensure all are moving freely. 2. Change pump tubing. 3. Perform BEC check. 4. Rinse spray chamber with dilute HNO_3 followed by D.I. H_20 when finished with last run of day. 5. Check to ensure gas supply is adequate. B. As Needed 1. Replace or clean nebulizer tips. 2. Disassemble torch, clean, and replace o-rings. 3. Perform wavelength calibration.	1. Torch 1. Torch 2. O-rings 3. Spray chamber 4. RF Coil 5. Nebulizer Tips 6. Pump tubing

TABLE 11-1

Instrument	Maintenance Procedures/Schedule	Spare Parts in Stock
Cold Vapor Atomic Absorption Spectrometer	1. Clean/inspect quartz cell daily. 2. Change pump tubing daily. 3. DI water rinse 10-15 minutes at end of run. 4. Check gas pressure daily. 5. Empty drain vessel daily.	1. Pump tubing 2. Quartz cell 3. Sampler probe
Lachat Autoanalyzer	1. Rinse manifold with degassed Milli-Q water before analysis. 2. Rinse manifold with recommended cleaning solution and degassed Milli-Q water after analysis. 3. Inspect pump tubings before analysis; replace if discolored or distorted. 4. Backup data files from computer onto a floppy disk every week. 5. Add water to heating block when necessary. 6. Clean work area daily.	1. Pump tubes 2. Quartz cell 3. Light bulbs
Spectrophotometer	1. Monitor and document absorbance for each analysis. 2. Rinse with DI water before and after analysis.	1. Light source bulbs
Ovens	1. Clean every month. 2. Monitor and document temperature daily.	1. Thermometer
pH Meter	1. Daily calibration. 2. Daily inspection of filling solution; add or replace when necessary.	1. Filling solution 2. pH buffers
Thermometers	1. Biannual calibration with an ASTM certified thermometer.	
Balances	1. Daily calibration with ASTM weights (S class). 2. Yearly calibration by an ASTM certified technician. 3. Daily cleaning.	
Gas Chromatograph GC VOCs	1. Change septa as needed. 2. Column maintenance as needed. 3. Clean injection port liner as needed.	1. Septa 2. GC columns 3. Syringes
Warzyn Field Instrumentation:		
pH Meter	1. Daily calibration. 2. Replace electrodes as needed. 3. Spare electrodes	1. pH buffers 2. Batteries
Conductivity Meter	1. Daily calibration. 2. Check redline and replace batteries if does not calibrate.	1. Batteries

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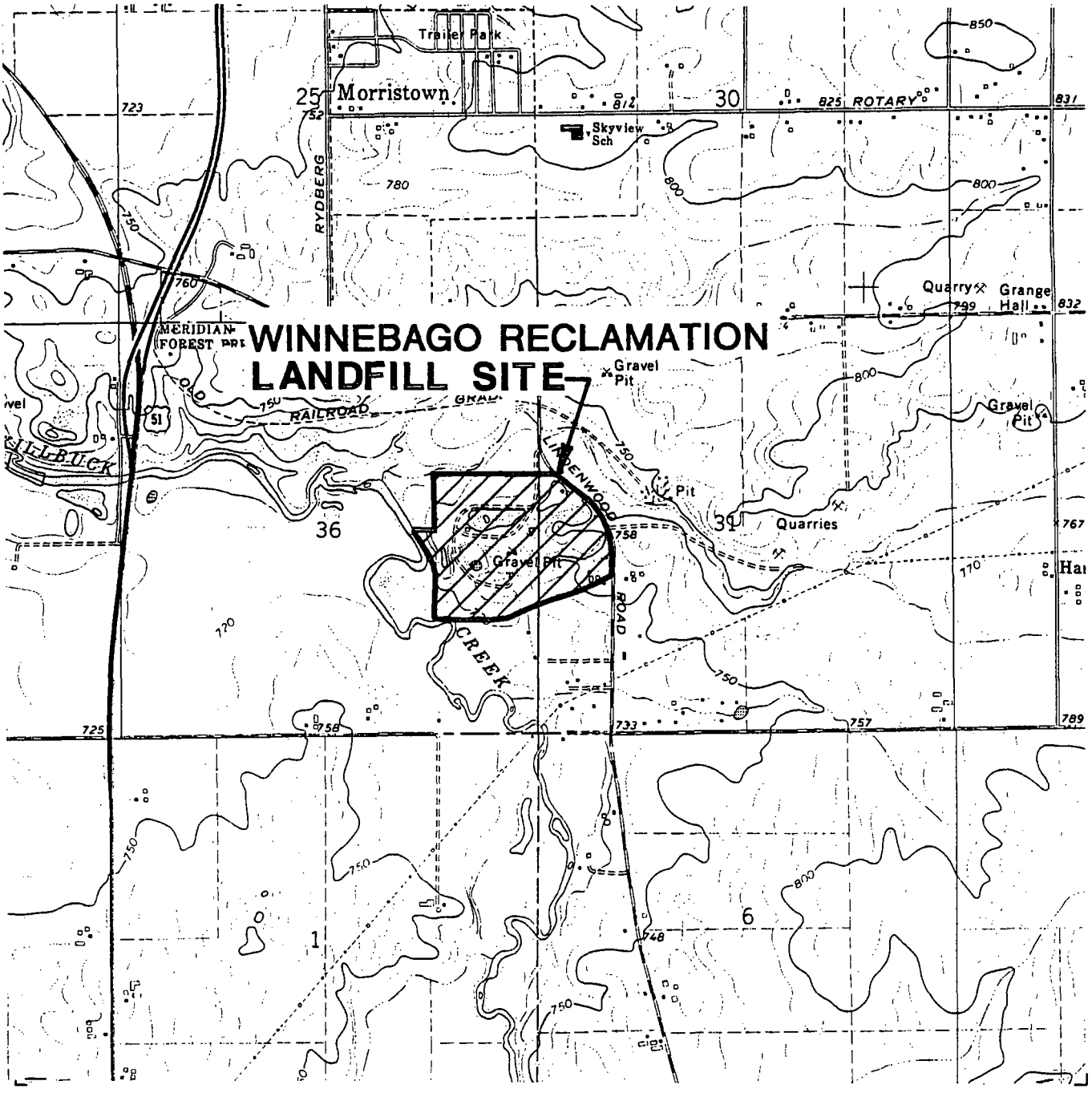
FIGURES



NOTES

1. BASE MAP DEVELOPED FROM THE ROCKFORD SOUTH, ILLINOIS 7.5 MINUTE USGS TOPOGRAPHIC QUADRANGLE MAP DATED 1971, PHOTOREVISED 1976.

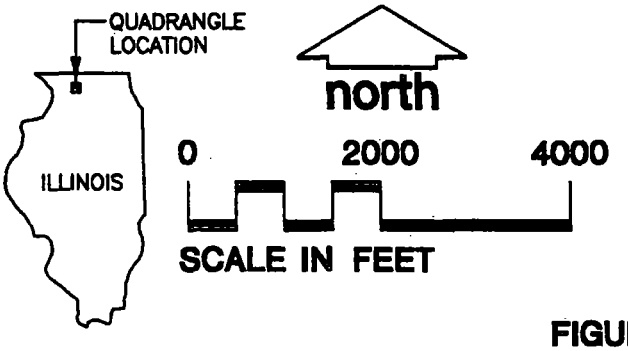


FIGURE 1-1

Developed By: CWB	Drawn By: ELR	
Approved By: Alan J. Schmidt	Date: 7/8/93	
Reference:		
Revisions:		

SITE LOCATION MAP

RD/RA QUALITY ASSURANCE PROJECT PLAN
WINNEBAGO RECLAMATION LANDFILL SITE
WINNEBAGO COUNTY, ILLINOIS

Drawing Number 10009721 **A4**

WARZYN

WARZYN INC.

QUALITY CONTROL
Graphic Standards Lead Professional
Technical Review Project Manager
Management Review Other

CATION AND NUMBER

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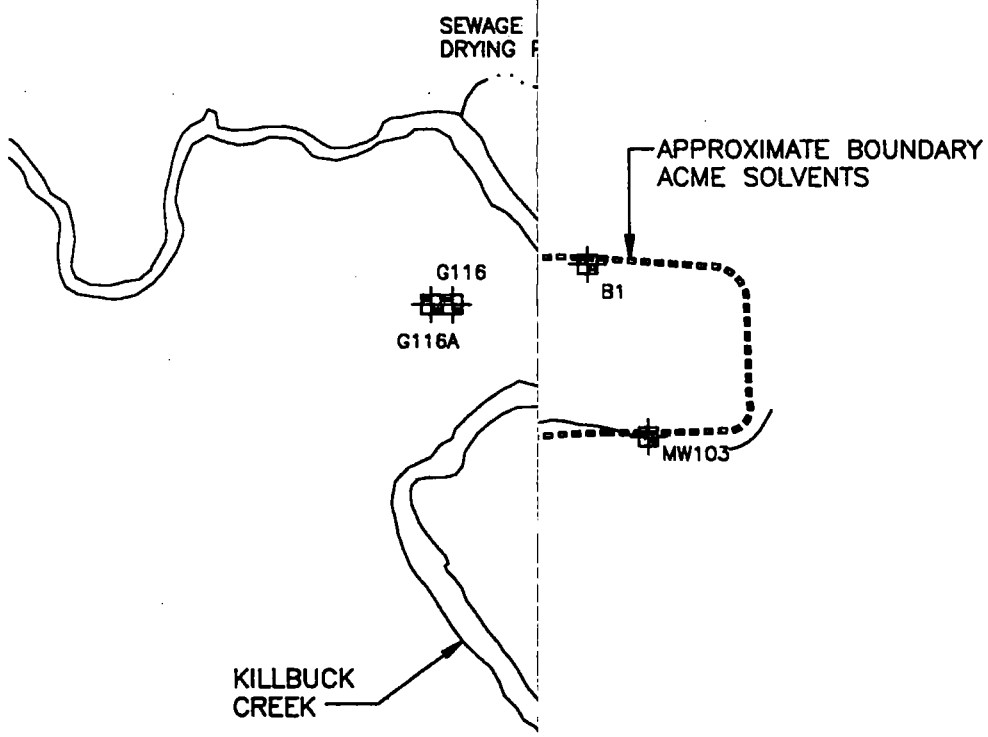
APPROXIMATE
WINNEBAGO R
LANDFILL SITE HOWN AS ONE SYMBOL



orth

600 1200

FEET

FIGURE 1-2

QUALITY CONTROL	Graphic Standards	Technical Review	Management Review
	Lead Professional	Project Manager	Other

Developed By: CWB Drawn By: ELR

Approved By: Date: 7/8/93

Reference:

Revisions:

SITE FEATURES MAP

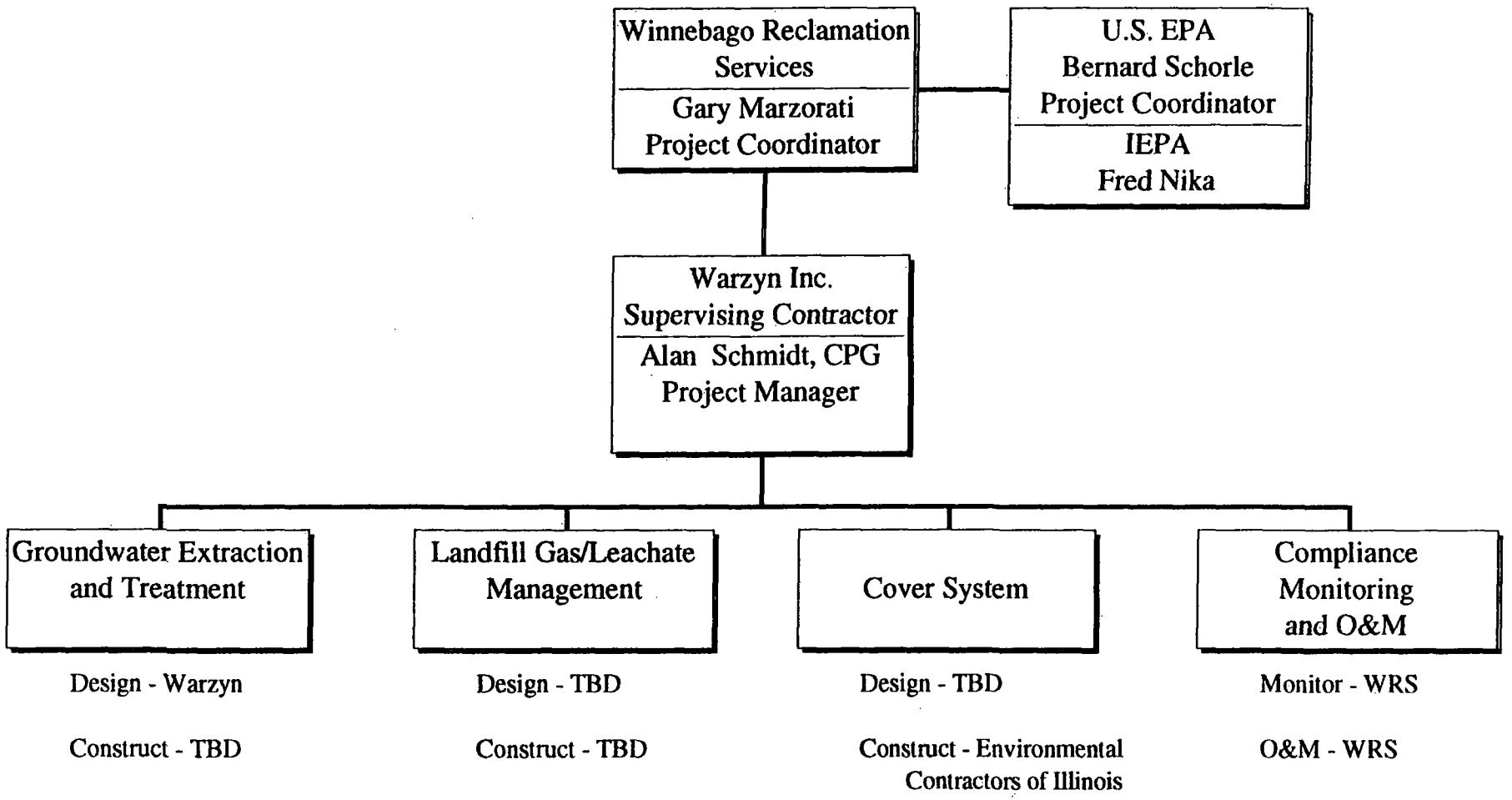
RD/RA QUALITY ASSURANCE PROJECT PLAN
WINNEBAGO RECLAMATION LANDFILL SITE
WINNEBAGO COUNTY, ILLINOIS

Drawing Number
10009721 B5

WARZYN

WARZYN INC.

Project Organization
Winnebago Reclamation Landfill RD/RA



TBD = To Be Determined

Figure 2-1

Project Organization
Winnebago Reclamation Landfill RD
Groundwater Extraction and Treatment

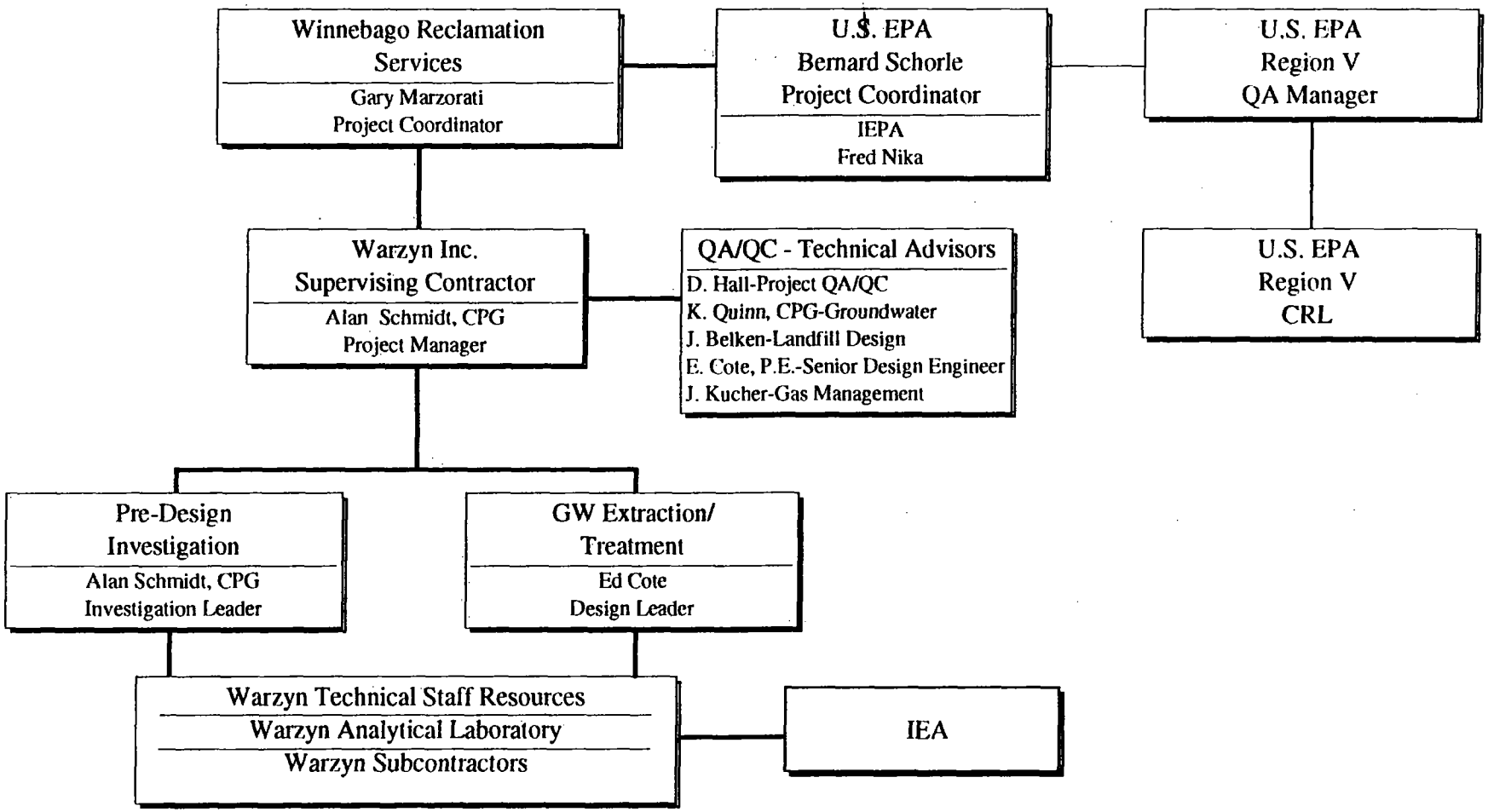


Figure 2-2

APPENDICES

A

A

SAMPLING PLAN

1

INTRODUCTION

This Sampling Plan (SP) for field activities describes the procedures to be used in collecting site investigation data for the Groundwater Extraction and Treatment System Remedial Design (Groundwater RD). The site investigation will be conducted in three phases.

Phase I

- Groundwater sampling and analysis of 19 wells along the western boundary

Phase II

- Groundwater and soil sampling and analysis during boring of deep wells
- Placement of two monitoring well nests in the southwest corner of the site
- Groundwater sampling and analysis of the new wells plus sampling of 3 to 6 selected existing wells

Phase III

- Installation of an extraction well
- Conducting a pumping test
- Groundwater sampling and analysis during the pump test

1.1 FIELD SAMPLING PLAN OBJECTIVES

Objectives of the proposed field activities are to:

- Establish the current quality of groundwater west (downgradient) of WRL through sampling and analysis of select existing monitoring wells
- Better define/confirm the horizontal and vertical extent of groundwater contamination west of the landfill by installing two well nests
- Define and confirm aquifer characteristics to be used in system design, by conducting a pumping test

The objective of the groundwater sampling phase of field activities is to determine the horizontal and vertical extent of contamination in the groundwater along the west boundary of the WRL Site. Wells and piezometers in that area will be sampled, and the results will be used to select the locations of two monitoring well nests to be installed west and south of monitoring well G115.

The second phase of field activities is to install the two monitoring well nests described above. The shallow well at each location will be a water table well. The groundwater in the deeper well will be sampled while drilling to define the vertical extent of contamination in the southwest corner of the site, and to select the optimum depth to place the well screen. The deep borings will be extended into materials which exhibit no detectable contamination. Wells will be screened in the contaminated zone based on analytical results. It is expected that wells will terminate in a sand and gravel aquifer which extends into a bedrock valley west of the site. If concentrations of contaminants are significant in the unconsolidated materials a third well may be added to the well nests to better monitor the effectiveness of the remedial system at depth within the unconsolidated sediments as well as bedrock. Information collected during the drilling and sampling of the two well nests will be used to optimize the screen locations of the groundwater extraction system wells in the southwest WRL Site area.

[mad-607-262q]

2

SCOPE

The Groundwater RD will consist of three phases: groundwater sampling of selected wells, placement of two well nests, and a pumping test.

Phase I Groundwater sampling will include:

- Sampling groundwater from 19 wells along the northwest, west, and southwest portions of the site
- Sample analysis
- Measuring water levels in those 19 wells and other site wells

Phase II Placement of two well nests will include:

- Construction of two well nests in the southwest corner of the site. Each nest will consist of a water table well and a deeper well, the depth to be determined by results of the purgeable GC VOC analysis.
- Collection of groundwater samples from respective intervals of the deeper borings using a single packer method.
- Collection of soil from the borings to be analyzed for grain size to aid in extraction well filter pack designs.
- Analysis of groundwater samples collected while drilling on a 24-hour turnaround basis (screening).
- Development and groundwater sampling of the newly installed wells and groundwater sampling of selected existing wells.

Phase III Pump Test will include:

- Installation of one extraction well to be used in the pumping test
- Performing a pumping test
- Collecting and analyzing groundwater samples during the test

[mad-607-262r]

3

SAMPLING LOCATIONS AND NUMBERS OF SAMPLES

3.1 PHASE I GROUNDWATER SAMPLING LOCATIONS

Phase I will include collecting a groundwater sample from each of the following 19 existing wells:

B10			
B10A	B15R		
B14	G118A	G116A	
B14A	G107	G117	MW106
B15P	G115	G118R	P1
	G116	G119	P3R
		G119A	P4R

Water level measurements will be collected in these and other existing monitoring wells. Samples will be analyzed for the following analytical parameters:

- TCL VOCs and SVOCs analyzed by Warzyn's Subcontract Laboratory, IEA in Triangle Park, North Carolina, using SOW OLMO1.8 with data validation
- Selected TAL metals and cyanide (Table 10, Groundwater RD Work Plan) analyzed by Warzyn Analytical Laboratory, Madison, Wisconsin with data validation using SOW ILMO3.0
- Indicator parameters (Table 10) analyzed by Warzyn Analytical Laboratory, Madison, Wisconsin using SOPs found in Appendix B of the QAPjP.

3.2 PHASE II MONITORING WELL NESTS

During Phase II, two monitoring well nests (G130 and G130A, G131 and G131A) will be installed to the west and south of monitoring well G115. Groundwater samples will be collected while drilling at approximately 20-ft intervals for purgable GC VOCs analysis in the deeper boring at each location. VOCs were selected for sampling during drilling because VOCs tend to migrate more quickly than other more conservative parameters, and chlorinated VOCs have been determined to be the primary compounds of concern at this site. The VOC samples will be analyzed on a 24-hr turnaround time and used for the purpose of determining screen placement for the well. The screen will be placed in the zone of highest VOC contamination. The deeper borings will terminate at a depth where VOCs are low or non-detectable.

Groundwater collected during drilling will be analyzed for the following analytical parameters:

- VOCs (Table A-1), analyzed by Warzyn's Analytical Laboratory (with no data validation) using methods indicated in the QAPjP

3.2.1 Groundwater Sampling

Each well in the newly installed well nests will be developed. Following development, the newly installed wells will be sampled. Additionally, the following five wells along the west boundary will be sampled.

- G115
- P3R
- P4R
- P1
- MW106

Samples will be analyzed for the analytical parameters using the analytical laboratories and methods stated in Section 3.1.

3.2.2 Soil Sampling

Two soil samples will be collected from each well nest location during drilling. A total of four soil samples will be collected and submitted to Warzyn's Geotechnical Laboratory for grain size analysis.

3.3 PHASE III GROUNDWATER SAMPLING DURING PUMPING TEST

During the pumping test approximately 8 groundwater samples will be collected and analyzed for TCL VOCs and SVOCs, selected TAL metals and cyanide, and indicator parameters. Groundwater samples will be analyzed by Warzyn's Analytical Laboratory and Subcontract Laboratory (with data validation) using methods indicated in the QAPjP.

3.4 QUALITY ASSURANCE SAMPLING

Quality Control samples will be collected during Phase I and Phase II sampling activities associated with the chemical characterization of groundwater. Quality control samples will consist of sample duplicates, field blanks, trip blanks, and matrix spike/matrix spike duplicate (MS/MSD) samples as described below.

3.4.1 Field Blanks

For water samples, one field blank will be prepared (for each type and container size). Field blanks will be prepared according to the following schedule for each sampling activity:

- One field blank for every 10 or fewer samples of groundwater collected
- For each sample period, a minimum of one blank for each group of parameters per sample matrix

The field blank samples will be prepared using deionized water stored in polyethylene containers. The water will be routed through the bailer or submersible pump for groundwater sample field blanks.

3.4.2 Trip Blanks (TB)

A trip blank for TCL VOC analysis will be included in each sample shipment container containing water VOC samples. The trip blank will consist of two 40-ml VOC vials filled with deionized water and preserved with 1+1 HCL. It will be prepared in the office or laboratory, transported to the field, and shipped with the other samples to the designated laboratory without being opened. It will be packaged using standard procedures as for other sample bottles.

3.4.3 Matrix Spike and Matrix Spike Duplicates (MS/MSD)

For water samples, one MS/MSD is required per group of 20 or fewer samples collected for VOC and SVOC analysis during each sampling activity. For TCL

VOCs, triple the normal sample volume will be collected (i.e., 12 40-ml VOC vials.

3.4.4 Sample Duplicate

One duplicate sample will be collected for each group of 10 or fewer samples collected for each matrix during each sampling period. A duplicate sample will consist of a sample obtained from the same sampling device as the original sample, by alternately filling the sample and duplicate bottles until the samples are collected.

[mad-607-262s]

4

SAMPLE DESIGNATION

A sample numbering system will be used to identify each investigative and quality control sample. Each sample identifier will include the project identifier code, sample type and location code, and a sampling event code. The sampler will maintain a log book containing the sample identification listings.

4.1 PROJECT IDENTIFIER CODE

A two-letter designation will be implemented to identify the sampling site. The project identifier will be "WRRD" to signify the Winnebago Reclamation Landfill Remedial Design investigation.

4.2 SAMPLE TYPE AND LOCATION CODE

Each sample collected will be identified by a two-letter code corresponding to the sample type. Sample type codes to be used for the subtasks covered in this sampling plan include:

- GW - groundwater sample from monitoring well
- FB - field blank
- TB - trip blank
- SB - soil sample from monitoring well boring

Other letter designators may be added for sample activities of later subtasks.

The location code will follow the sample type code. The location code consists of a two- to five-digit numeric or alpha-numeric code that indicates the sample location. Field blanks and trip blanks will use a consecutive numbering system starting at 01, assigned in the field.

4.3 SAMPLING ROUND CODE/DUPLICATE CODE

A two or three-digit numerical code will be used to designate additional location information. For groundwater and soil samples collected while drilling (Phase II), the Round code will represent the depth of the sample in feet below the ground surface. Duplicate samples will be designated by the Round code preceded by a 9. Matrix spike and matrix spike duplicate samples are collected as additional sample volume at selected locations.

Although identified as such on chain-of-custody records, specific sampling codes will not be provided for matrix spikes or matrix spike duplicates samples.

4.4 EXAMPLES OF SAMPLE NUMBERS

Examples of sample number codes are as follows:

- WRRD-GWG116A-01 = Winnebago Reclamation Landfill Remedial Design, groundwater sample from monitoring well G116A, Round 1
- WRRD-GWG116A-91 = Winnebago Reclamation Landfill Remedial Design, duplicate groundwater sample from monitoring well G116A
- WRRD GWG123A-30/42 = Winnebago Reclamation Landfill Remedial Design, groundwater sample collected from boring G123A using single packer method at a depth of 30 to 42 feet

[mad-607-262t]

5

GENERAL SAMPLING EQUIPMENT PROCEDURES

5.1 PHASE I INVESTIGATION

5.1.1 Groundwater Sampling

One round of groundwater samples will be collected from the following 19 wells:

B10	B15R	G116A	MW106
B10A	G118A	G117	P1
B14	G107	G118R	P3R
B14A	G115	G119	P4R
B15P	G116	G119A	

Groundwater elevations will be measured in the above wells and other existing WRL Site monitoring wells.

The procedure for groundwater sampling is described below. A groundwater level measurement will be taken using a decontaminated electric water level indicator. Three to five well volumes of water will be removed from the well prior to sampling using a clean bailer and cable or submersible pump. If a pump is used, the pump will be decontaminated between wells by pumping a soap solution followed by deionized water through the pump and tubing and washing the outside tubing which comes in contact with well water. The sample will be collected using a stainless steel bailer attached to stainless steel cable. Samples will be transferred from the bailer directly to the appropriate sample containers. Samples will be filtered and preserved (as described in Table 1-2 of the QAPjP) in the field, within 15 minutes of sample collection, and field parameters will be analyzed as soon as possible after sample collection. Samples will be kept on ice in coolers and shipped to the analytical laboratory at the end of each day. Well

locations are shown in the Groundwater RD Work Plan. The samples will be collected and analyzed as specified in Section 7.

5.1.2 Water Level Measurements

Groundwater levels will be measured at identified WRL Site wells during Phase I activities. Measurements will be taken using an electric water level indicator.

5.2 PHASE II INVESTIGATION

5.2.1 Drill Boring(s)/Install Monitoring Wells

The procedures used to drill borings are described in the following paragraphs.

Borings will be advanced through the unsaturated zone using a rotary drill with temporary casing and drilling mud or clean water. Drilling mud will not have an adverse effect on sampling to be conducted while drilling.

After entering the saturated zone, groundwater samples will be collected during the drilling at intervals of approximately 20 feet. Two soil samples will be collected from each well nest location and submitted for grain size analysis.

A packer system will be inserted into the temporary steel well casing and the packer will be inflated to test the system. If the packer inflates properly and seats against the casing, the packer will be deflated and the system will be lowered to the appropriate sampling depth. The packer system will be reinflated to isolate a 20-foot long interval of the borehole. A submersible pump will be lowered down the steel tubing attached to the packer system to a point just below the packer. Two to three borehole volumes of water will be removed prior to sampling, if possible (the amount removed will depend on the rate that the borehole will produce water).

A groundwater sample will be collected in 40-ml. VOC vials directly from the discharge line of the submersible pump and analyzed for VOCs for screening purposes to determine the zone of highest VOC contamination in the aquifer. The well screen will be installed in the zone of highest VOC contamination. A low discharge rate will be used to minimize the potential for volatilization of contaminants. The sample will be kept on ice and maintained under standard chain-of-custody procedures. Samples will be shipped via overnight express courier to Warzyn's Analytical Laboratory in Madison, Wisconsin. Sample will be analyzed within 24 hours of sample collection.

The submersible pump will be decontaminated between sample intervals by pumping a soap solution followed by deionized water through the pump and

associated tubing. The packer system will be decontaminated by steam cleaning between sample intervals. The groundwater at these wells will be analyzed for the parameters specified in Section 7 using the procedures specified in the RD/RA Quality Assurance Project Plan.

Wells will be constructed of stainless steel screen (5 to 10 ft nominal depending on the vertical dimensions of the contaminated zone) and steel riser pipe. Wells will be constructed with reference to 77 IAC 920 Illinois Water Well Construction Code. The boring will be backfilled with No. 30 flint sand to approximately two feet above the top of the well screen. One to two feet of fine silica sand will be placed above the sandpack to prevent migration of the bentonite seal into the sandpack. A 2 ft bentonite chip seal will be placed above the fine sand. The remainder of the borehole annulus will be backfilled with thick bentonite slurry. A protective locking casing will be installed at ground surface. Granular bentonite will be placed around the protective casing.

The wells will be developed, after construction, using a clean bailer or submersible pump. A minimum of 10 well volumes will be purged and containerized. Purge water will be disposed of in the WRL leachate treatment system. Specific conductivity and pH measurements will be measured during well development to assess well stabilization. Development will continue until pH and specific conductance have stabilized.

The wells will be surveyed for location (+/- 1 ft) and elevation (+/- 0.01 ft). Drilling equipment will be decontaminated in accordance with the procedures in Section 6.

5.3 PHASE III INVESTIGATION

An extraction well will be installed and equipped with a submersible pump. Groundwater quality samples will be collected from the extraction well during the pump test and will be analyzed for TCL VOCs and SVOCs selected TAL metals and cyanide, and indicator parameters (Table 10, Groundwater RD Work Plan) to assess treatment system requirements. It is anticipated that approximately 8 samples will be collected and analyzed using procedures stated in Section 7.

[mad-607-262u]

6

DECONTAMINATION PROCEDURES

Procedures to be followed to decontaminate equipment and personnel are described in the RD/RA Site Health and Safety Plan. The procedures are summarized below.

6.1 SITE PERSONNEL DECONTAMINATION PROCEDURE

- Dispose of outer latex booties
- Wash boots in Liquinox bootwash
- Clean outer gloves in Liquinox wash solution (discard if too soiled to clean thoroughly)
- Dispose of polycoated tyvek suits
- Dispose of surgical gloves
- Wash hands in hand wash
- Wash face and neck in face wash
- Clean and sanitize face mask

Site personnel will perform the above mentioned decontamination procedure prior to leaving the site.

Discarded clothing and other articles will be collected in double-lined, heavy duty garbage bags.

6.2 EQUIPMENT AND VEHICLE DECONTAMINATION PROCEDURE

- Decontamination will be performed prior to site entry
- Decontamination will be performed on-Site
- Gross contamination will be removed with a brush and Liquinox solution
- Steam cleaning or hot water high pressure washing will follow

The drilling equipment and the backhoe will be steam cleaned or high pressure hot water washed between boring/excavation locations at the borehole/excavation prior to exiting the site. Another area, located away from the equipment decon area, will be designated as a site personnel decon area.

Decontamination will include steam-cleaning or hot water high pressure washing the drilling equipment, and tools between boreholes and detergent washing and water rinsing the split spoon samplers after each collected sample. The drill rig and equipment may also require scrubbing of accessible parts with a detergent/water solution. Well materials will also be steam-cleaned or hot water high pressure washed and wrapped in plastic until installed. The bailer cable and water level measurement tape will be cleaned with Liquinox or another non-phosphate detergent solution, and rinsed with deionized water.

Potentially contaminated equipment remaining on-Site overnight, and in areas accessible to the public, will be decontaminated by the end of the day.

Decontamination water will be contained and disposed of in the WRL leachate treatment system.

[mad-607-262v]

7

SAMPLE HANDLING AND ANALYSIS

7.1 PARAMETERS AND METHODS

Groundwater samples collected from specified existing and new wells during Phase I, II and III activities will be analyzed for the chemical parameters specified in Table 10 of the Groundwater RD Work Plan using the following laboratories and methods.

- TCL VOCs, and SVOCs analyzed by Warzyn's subcontract Laboratory IEA in Triangle Park, North Carolina using CLP SOW OLMO1.8 (or most current).
- TAL selected metals and cyanide are analyzed by Warzyn using CLP SOW ILMO3.0 (or most current)
- Purgeable VOCs by GC for screening purposes and indicators are analyzed by Warzyn Analytical Laboratory, Madison, Wisconsin using SOPs found in Appendix B of the QAPjP.

Measurement of pH, specific conductance, and temperature will be performed in the field.

Groundwater samples collected from borings using the packer method during Phase II will be analyzed for the parameters listed in Table A-1 of this Sampling Plan by Warzyn Analytical Laboratory, Madison, Wisconsin using Methods 8010/8020 (no data validation). This analysis is for screening purposes, only, to determine the zone of highest VOC contaminants where the well screen

will be placed.

7.2 SAMPLE PRESERVATION

VOC samples will be collected with either a stainless steel bailer or a submersible pump after three to five well volumes have been purged. VOC samples are collected immediately upon completion of purging. VOC samples are collected with no headspace and preserved with 1+1 HCL.

Samples will be collected and preserved in a manner appropriate for the analyses they receive (see Table 1-2 of the RD/RA QAPjP). The metals and TDS portion of the sample requiring field filtering prior to analysis (see Table 1-2 of the RD/RA QAPjP) will be filtered, using a pressure filtration device, through a 0.45 micron filter within 15 minutes after collection. Filtered portions of the sample, field blank, and field duplicate will be preserved, as appropriate, for metals analysis, immediately after filtration. Preservatives added to the samples will be prepared using reagent grade chemicals. Table 1-2 of the RD/RA QAPjP should be consulted for details regarding sample packaging and shipping. Non-filtered metal samples will not be collected.

Indicator parameters are not filtered prior to analyses. Indicators will be collected in this order: TSS, alkalinity, sulfate, chloride, BOD, COD, TOC and ammonia nitrogen.

7.3 SAMPLE CONTAINER REQUIREMENTS

Contaminant-free sample containers (bottles) will be used for analyzing VOCs, metals and indicators for this sampling effort. The bottles will be prepared according to the procedures specified in U.S. EPA's "Specifications and Guidance for Obtaining Contaminant-Free Sample Containers, April 1990". Bottles used for sampling will not contain target organic and inorganic contaminants exceeding the level specified in the above mentioned document. Specifications for the bottles will be verified by checking the supplier's certified statement and analytical results for each bottle lot, and will be documented on a continuing basis. This data will be maintained in the project evidence file and will be available, if required, for EPA review.

In addition, the data for field blanks, rinsate blanks, and trip blanks, etc., will be monitored for contamination, and corrective actions will be taken if a problem is identified, to determine whether the analytical data is usable. This corrective action may include discontinuing the use of a specific bottle lot, contacting the

bottle supplier(s) for retesting the representative bottle from a suspect lot, re-sampling the suspected samples, validating the data taking into account that the contaminants could be introduced by the laboratory (i.e., common lab solvents, sample handling artifacts, etc.) or could be bottle QC problem.

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8

SAMPLE DOCUMENTATION

Field sampling activities will be documented using a bound notebook/logbook. Information recorded in the field notebook will include date of sampling, sampler, weather conditions, observations and methods of preservation. Additional data pertaining to sampling may also be included in the logbook.

Samples will be collected under chain-of-custody procedures. Standard forms including sample labels, sample tags, chain-of-custody forms, and custody seals used for sample tracking will be maintained (see attachments). A brief description of sample documents follow:

A. Chain of Custody Form

1. One Form per shipping container (cooler).
2. Carrier service does not need to sign form, if custody seals remain intact.
3. Use for all samples.

B. Chain of Custody Seals

1. Two seals per shipping container to secure the lid and provide evidence that samples have not been tampered with.
2. Cover seals with clear tape.
3. Record seal numbers on Chain of Custody Form.
4. Use for all samples.

C. Sample Tags

1. Each sample container must have a sample tag affixed to it.
2. Sample tag numbers are recorded on the Chain of Custody Forms.
3. Use for all samples.

D. Sample Identification Record Form:

1. Provide means of recording crucial sample shipping and tracking information.

2. Contain information such as:

- Sample number
- Sample matrix
- Sample location code
- Sample round
- Chain of custody number
- Lab code
- Date sampled
- Date shipped
- Airbill number
- Sampling tag number

Paperwork accompanying the samples being shipped to the laboratory will be sealed in a plastic bag that is taped to the inside of the cooler lid. Copies of the chain-of-custody forms, and other paperwork (if possible), will be retained with the field files.

Two sample seals will be placed on opposite sides of the lid and extending down the sides of the cooler. The lid will be securely taped shut prior to shipment.

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B

B

ANALYTICAL SERVICES STANDARD OPERATING PROCEDURES

(SOPs)

B1 Purgeable VOCs
B2 Total Alkalinity
B3 Chloride
B4 Sulfate
B5 Ammonia Nitrogen
B6 Total Suspended Solids (TSS)
B7 Total Dissolved Solids (TDS)
B8 Total Organic Carbon (TOC
B9 Chemical Oxygen Demand
B10 Biochemical Oxygen Demand (BOD_5) and YSI Dissolved Oxygen (DO)
B11 Nitrate & Nitrite Nitrogen

B1

PURGEABLE VOCs

PURGEABLE VOLATILE ORGANICS

Scope and Application: This method covers the determination of volatile organic compounds in water and soils. Various compounds can be analyzed using this method. Refer to Table 1 for a breakdown of some common compound combination requests and their associated reporting limits.

Compounds:

Benzene
Bromodichloromethane
Bromoform
Bromomethane
Carbon tetrachloride
Chlorobenzene
Dibromochloromethane
Chloroethane
2-Chloroethylvinyl ether
Chloroform
Chloromethane
Ethylbenzene
Bromobenzene
sec-Butylbenzene
4-Chlorotoluene
Dichlorodifluoromethane
1,1-Dichloropropene
p-Isopropyltoluene
Styrene
1,2,4-Trichlorobenzene
1,3,5-Trimethylbenzene
Acrolein
Methyl ethyl ketone(MEK)
Methyl tert-butyl ether(MTBE)
tert-Butyl alcohol
1,2-Dichlorobenzene
1,3-Dichlorobenzene
1,4-Dichlorobenzene
1,1-Dichloroethane
1,2-Dichloroethane
1,1-Dichloroethene
cis-1,2-Dichloroethene
trans-1,2-Dichloroethene
1,2-Dichloropropane
cis-1,3-Dichloropropene
trans-1,3-Dichloropropene
Methylene chloride
Bromochloromethane
tert-Butylbenzene
1,2-Dibromoethane
1,3-Dichloropropane
Hexachlorobutadiene
Naphthalene
1,1,1,2-Tetrachloroethane
1,2,3-Trichloropropane
Acetone
Acrylonitrile
Methyl isobutyl ketone(MIBK)
1,1,2,2-Tetrachloroethane
Tetrachloroethene
Toluene
1,1,1-Trichloroethane
1,1,2-Trichloroethane
Trichloroethene
Trichlorofluoromethane
Vinyl chloride
m-Xylene
o-Xylene
p-Xylene
Di-isopropyl ether
n-Butylbenzene
2-Chlorotoluene
Dibromomethane
2,2-Dichloropropane
Isopropylbenzene(Cumene)
n-Propylbenzene
1,2,3-Trichlorobenzene
1,2,4-Trimethylbenzene
Tetrahydrofuran
Trichlorotrifluoromethane
1,2-Dibromo-3-chloropropane

Notes:

1. m-Xylene and p-Xylene are not resolved on the VOCOL column and will be reported as the sum of the unresolved set. Separation and confirmation will be performed only when requested.

2. tert-Butyl alcohol can be analyzed using this method via an FID detector.

Method Reference:

"Methods for Organic Chemical Analysis of Municipal and Industrial Wastewater", July 1982, Methods 601, Purgeable Halocarbons, and 602, Purgeable Aromatics.

"Test Methods for Evaluating Solid Waste", EPA-SW846, September 1986, Methods 8010, Halogenated Volatile Organics and 8020, Aromatic Volatile Organics.

"Volatile Organic Compounds in Water by Purge and Trap Capillary Column Gas Chromatography with Photoionization and Electrolytic Conductivity Detectors in Series", September 1986, EPA Method 502.2.

Reporting Limits:

Waters: 1.0 - 20 ug/L (See Table 1)
Low Level Soils: 5.0 - 100 ug/kg; 1.0 ug/kg for Illinois samples (See Table 1)
Medium/High Level Soils: 50 - 1000 ug/kg (See Table 1)

Optimum Range:

Up to 100 times the reporting limit for each matrix group.

Sample Handling:

Waters: Water samples are to be collected in duplicate in 40 mL glass vials with open screw-caps and teflon faced silicone septa. They must be collected with no headspace in the bottle. Samples should be preserved by adding two drops of 1:1 HCl. All samples must be refrigerated at 4°C and analyzed within 14 days of collection. Note: If samples are not preserved, samples must be refrigerated at 4°C and analyzed within 7 days of collection.

Soils: Soils should be collected in 4 oz glass jars with teflon lined caps. Samples should be packed tightly so that there is no headspace or air pockets in the sample. All samples must be refrigerated at 4°C and analyzed within 14 days of collection.

Reagents and Apparatus:

1. Open screw cap 40 mL vial (Pierce #13075 or equivalent).
2. Septum - teflon-faced silicone (Pierce #12722 or equivalent).
3. Purge and trap device - Tekmar LSC2000 equipped with ALS2016 multiple sample option.
4. Trap-12" long with 3" Tenax, 3" Silica Gel, 3" Coconut Charcoal, Tekmar Part # 14-0124-003.
5. Purge tubes - 5 mL fritted, and 25 mL disposable culture tubes (3/4" O.D.)

6. Gas chromatograph - Varian 3400 equipped with PID and Hall detectors in series, megabore capillary column adapters, makeup gas, and cryogenics.
7. Column - 60 m x 0.75 mm I.D., 1.5 um film thickness; VOCOL megabore column, Supelco Part # 2-373M (105 m x 0.53 mm I.D. Rtx-502.2, Restek Part # 10910 for only those samples which require tert-Butyl Alcohol).
8. Glass Syringes:
   5 mL Leurlock tip
   1 mL Hamilton
   100 uL Hamilton
   10 uL Hamilton
   25 uL Hamilton
9. Balance - $\pm$0.0001 g (Mettler AE200)
10. Balance - $\pm$0.01 g (Sartorius, 1202 MP)
11. Reagent water - water which has been shown to be organic-free at the method detection limits (tap water).
12. Methanol - B&J Brand Purge and Trap Grade
13. Beakers - assorted
14. Volumetric flasks - assorted
15. Pipettes - 5 and 10 mL volumetric
16. Mininert Valves - Leurlock
17. Helium
18. Hydrogen - ulta high purity grade
19. Liquid CO_2
20. Standards (refer to reagent preparation section)

Note: Apparatus can be substituted with equivalents.

<u>Gas Chromatographic Conditions</u>:

Column:

60 m x 0.75 mm I.D., 1.5 um film thickness; VOCOL megabore column (105 m x 0.53 mm I.D. Rtx-502.2, Restek Part # 10910 for only those samples which require tert-Butyl Alcohol).

Carrier Gas:

Helium
10 mL/min, make up to 35 mL/min

Detectors (in series):

1. Photo Ionization - 10.0 eV
   Sensitivity - Range 11 x Attenuation 8
   Temperature - 250°C

2. Electrolytic Conductivity - Hall 1000 or Hall 700A/ECD
   Mode - Halogen

Reactor temperature - 900°C
Solvent Flow - 0.5 mL/min Methanol
Hydrogen Flow - 50 mL/min

Oven:

Initial - 5°C, Hold for 8 min
Ramp - 4°C/min
Final - 180°C, Hold for 2 min

Purge & Trap System:

Trap: Tenax/Silica Gel/Coconut Charcoal
Purge Flow:
Aqueous samples- 40 mL/min
Non-aqueous samples- 60 mL/min for Soils
Purge Time: 5 minutes
Dry Time: 0 minutes
Desorb: 4 minutes at 180°C
Bake: 15 minutes at 225°C
Transfer line: 100°C

Note: Analysis conditions may be changed to optimize the analysis provided that sensitivity and resolution is determined to be equal to or better than initial demonstration of capabilities.

Reagent Preparation:

1. Stock standard preparation guidelines are provided as Attachment I.
2. Working standard preparation guidelines are provided as Attachment II.

Procedure:

Preparation of Calibration Standards:

The instrument must be calibrated for all compounds of interest. Follow the chart below for calibration standard preparation:

Standard	Concentration	Volume of Working Stock Standard to 100 mL reagent water
STD1	1.00 ug/L	5 uL
STD2	2.00 ug/L	10 uL
STD3	5.00 ug/L	25 uL
STD4	10.0 ug/L	50 uL
STD5	20.0 ug/L	100 uL
STD6	40.0 ug/L	200 uL

Analysis of Waters:

1. Before any analyses can be performed, all purging vessels must be cleaned with reagent water at least 3 times. For extremely dirty vessels, clean them with a 1:10 HCl solutions, rinse with reagent water 3 times and dry them in an oven overnight.

2. Remove the plunger from a 5 mL leurlock syringe. Fill the syringe with the low level standard. Replace the plunger and adjust the volume to 5 mL, making sure that air bubbles are not present. Load onto autosampler. **Repeat this step for all of the calibration standards, from low concentration to high.** Note: If the 502.2 list is required, add 5 uL of the 502.2 surrogate solution (10 ug/mL) to each of the calibration standards. The average response is calculated, and the surrogate recoveries are calculated against this average response.

3. After the calibration standards have been loaded and analyzed, the QC check standard must be analyzed. Fill a 5 mL syringe with the working check standard, and load onto the ALS.

4. Prepare a method blank by filling the 5 mL syringe with 5 mL reagent water. Add 5 uL of the 601/602 surrogate (10 ug/mL) and 5 uL of the 502.2 surrogate (10 ug/mL) solutions through the valve bore of the syringe. Load onto autosampler.

5. If all criteria is met for the initial calibration, the QC check standard, and the blank, samples can be loaded onto the instrument and analyzed.

6. Prepare a laboratory fortified blank (LFB) by filling a 5 mL syringe with 5 mLs of reagent water. Add 5 uL of the 10 ug/mL working matrix spike solution, 5 uL of the 10 ug/mL 601/602 surrogate solution, and 5 uL of the 10 ug/mL 502.2 surrogate solution. Load onto ALS.

7. Samples are prepared by filling the 5 mL syringe with 5 mL of sample, and add 5 uL of the either the 10 ug/mL 601/602 surrogate solution or the 10 ug/mL 502.2 surrogate solution, depending upon the analysis requested. Load onto the ALS. **Note: This results in a final surrogate concentration of 10 ug/L.**

8. After every 10 samples analyzed, a matrix spike and matrix spike duplicate must be analyzed. These are prepared like regular samples <u>except</u> that 5 uL of the 10 mg/mL working matrix spike solution is added to the 5 mL of sample. This should be performed in duplicate on the same sample. Do not spike trip or field blanks. Load them onto the autosampler. **Note: This results in a final concentration of 10 ug/L for each of the matrix spike analytes.**

9. At the end of an analytical run, a 10 ppb continuing calibration verification (CCV) standard must be analyzed. Prepare a 10 ug/L CCV standard outlined in the table in the "**Preparation of calibration standards**" section. For compounds with elevated reporting limits, a CCV standard is run at 10 times the concentration of the reporting limit.

10. If the sample response for any of the compounds is greater than the response of the highest calibration standard for any analyte, dilution is required. The level of the dilution should be such that the concentration of the analyte(s) is (are) approximately mid-range in the calibration.

The run order should be as follows:

Day 1

1. 1.00 ug/L calibration standard
2. 2.00 ug/L calibration standard
3. 5.00 ug/L calibration standard
4. 10.0 ug/L calibration standard
5. 20.0 ug/L calibration standard
6. 40.0 ug/L calibration standard
7. QC Check Standard (to verify initial calibration)
8. Method Blank
9. Laboratory fortified blank
10. Ten samples
11. Matrix spike
12. Matrix spike duplicate
13. **Repeat sequence 10-12.**
14. End the run with the analysis of a continuing calibration verification standard (CCV).

Day 2 (and succeeding days)

1. Analyze a CCV standard.
2. Analyze a method blank.
3. Proceed with sequence #10 from Day 1.

Analysis of Low Level Soils:

1. This analysis is performed on soil samples which contain levels of contamination less than 100 ug/kg or when lower detection limits are required.

2. Calibration: Follow the same procedure for calibrating the instrument as for the water analysis.

3. Samples are prepared by weighing 1-5 $\pm$ 0.03 grams of soil (amount depends on desired detection limit) into the purging vessel. Record the weight, and load the purge vessel onto the

autosampler.

4. Add 5 mL of reagent water spiked with 5 uL of the 10 ug/mL working surrogate to the purge vessel containing the soil sample. Note: For samlpes requiring 5 grams of soil, 10 mL of reagent water is added fof better purging.

5. Matrix spikes and matrix spike duplicates are analyzed every 10 samples as with the water analysis. Add 5 mL of reagent water spiked with 5 uL of the 10 ug/mL working matrix spike and 5 uL of the 10 ug/mL working surrogate to the purge vessel containing a preweighed amount of soil sample. This should be done in duplicate on the same sample.

6. At the end of every run a 10 ug/L continuing calibrations standard must be analyzed.

Analysis of Medium/High Level Soils:

1. This analysis is performed on soil samples which contain levels of contamination above 100 ug/kg or when low detection limits are not required.

2. The calibration, QC check standard, and method blank are analyzed the same as for the water analysis.

3. A methanol blank must be analyzed. This is prepared by adding 100 uL of methanol to 5 mL of reagent water, and 5 uL of each of the 10 ug/mL 601/602 and 502.2 working surrogates, and then loaded onto the autosampler.

4. Samples are prepared by weighing 5.0 $\pm$ 0.01 g of soil in a 20 x 50 mm screw cap centrifuge tube. Record the weight. Add 5 mL of methanol and 12.5 uL of the 500 ug/mL working surrogate. Cap with a teflon lined screw cap, and invert 20 to 30 times (clay samples may require more inverting). Allow the extract to settle.

5. Add 100 uL of the extract to 5 mL of reagent water which contains 5 uL of the 10 ug/mL working soil surrogate. Load onto the autosampler.

w the chart below if other dilutions are need in order for sample concentrations to fall
de the established calibration range.

ation	Weight Sample	mL Methanol	Surrogate	uL Extract Added to 5 mL Reagent Water
X	5.00 g	5.00 mL	12.5 uL of 500 ug/mL	100 uL
00X	5.00 g	5.00 mL	25.0 uL of 500 ug/mL	50 uL
250X	1.00 g	5.00 mL	12.5 uL of 500 ug/mL	100 uL
500X	1.00 g	5.00 mL	25.0 uL of 500 ug/mL	50 uL
1000X	1.00 g	5.00 mL	50.0 uL of 500 ug/mL	25 uL
2500X	1.00 g	5.00 mL	12.5 uL of 5000 ug/mL	10 uL

7. Matrix spikes and matrix spike duplicates should should be analyzed after every 10 samples analyzed. Prepare the matrix spike, in duplicate, by adding 20 uL of the 250 ug/mL stock matrix spike solution to the preweighed soil sample containing methanol. Follow steps 4 and 5 of High levels soils for loading.

8. At the end of every run, a 10 ug/L continuing calibration standard must be analyzed, making sure to add 100 uL of methanol to the 5 mL of standard.

Quality Control:

1. **QC Check Standard:** After every calibration curve analyzed, a check standard (prepared independently of calibration standards) must be analyzed (at 10 ppb). All analytes must fall within ±30% of the concentration (7-13 ppb) for the system to be considered in control. For gases (chloromethane, chloroethane, bromomethane, vinyl chloride, dichlorodifluoromethane, trichlorofluoromethane, and trichlorotrifluoromethane) and 2-chloroethylvinyl ether, the limits are ±50% (5-15 ppb). If this criteria is not met, corrective action must be taken before samples can be analyzed.

2. **Method Blank:** A method blank must be analyzed at the beginning of every working day before any samples are analyzed. All compounds of interest must be less than the detection limit. If any compounds are found in the blank above the detection limit, corrective action must be taken to alleviate the contamination. Any samples analyzed with the problem blank must have the detected compounds flagged on the final report.

3. **Matrix Spike/Matrix Spike Duplicate:** For each type of matrix, a minimum of 10% of the samples must be spiked in duplicate. The percent recoveries and relative percent difference (%RPD) for each of the spiked compounds must be within the laboratory's established control limits. If criteria is not met, the spikes must be repeated.

If the control limits are not met a second time, corrective action needs to be taken.

4. **Laboratory Fortified Blank (LFB):** For every batch of samples analyzed, one LFB must be prepared and analyzed. If the batch contains more than 15 samples, then an LFB must be analyzed for every 15 samples or fraction thereof. The intent of this sample is to demonstrate the ability of the laboratory to provide acceptable results in the absence of sample matrix effects. In the event that poor recoveries are obtained for batch sample MS or MSDs, the LFB is evaluated to verify that the recoveries associated with the MS/MSD are matrix related, and the overall laboratory performance is not in question. The same control limits as for MS/MSDs are applied to LFB samples.

   If, however, acceptable results cannot be obtained for the LFB, the problem must be identified and resolved prior to the analysis of samples. The data from LFB samples are maintained on a control chart.

5. **Surrogates:** Working surrogates are added to each sample to monitor the performance of the method. Surrogate recoveries are calculated on an external standard basis. If a surrogate recovery falls outside of the acceptable control limits, the sample should be reanalyzed. If the surrogate recovery still falls outside of the control limits, sample results should be flagged.

6. **Initial Calibration Criteria:**
   - A minimum of 5 calibration levels must be included in the curve. One of these levels must be at 1 ppb.
   - All 5 levels chosen for the linear regression must be the same for every compound.
   - The correlation coefficient (r) for the calibration curve must be ≥ 0.995 (for Waters software, $r^2 \geq 0.990$).
   - The x-intercept (For Water's software, |A|/B) expressed as concentration, must be less than the reported detection limit for each analyte (1.0 ppb for all except: 2.0 ppb for m+p-xylene, dichlorodifluoromethane, 1,2-dibromoethane, chloromethane, bromomethane, and chloroethane; 3.0 ppb for methylene chloride and 1,2-dibromo-3-chloropropane; 5 ppb for MEK, MIBK, acrolein, and acrylonitrile; and 20 ppb for acetone and tetrahydrofuran).
   - If any of the above criteria is not met, corrective action must be taken, and the calibration standards analyzed again.

7. **Continuing Calibration Verification (CCV) acceptance criteria:**
   - The initial calibration must be verified each working day before any samples can be analyzed.

- A CCV at 10 ppb must be analyzed at the beginning and end of every analytical run. For compounds with elevated reporting limits, a CCV at a concentration of 10 times the concentration of the reporting limit is analyzed.
- The recoveries for all compounds but gases must be within the 70-130% criteria. Gases are to be within 50-150% criteria. If any analyte fails to meet these criteria, then the following conditions must be applied:
  - A calibration standard at 1.0 ppb (or at the reporting limit when required) is analyzed to verify that detection of the analyte can be achieved, with recoveries for the affected analytes within 70-130% (50-150% for gases and 2CLEVE).
  - The affected analytes do not fail the criteria in 2 consecutive CCVs (to ensure that the problem is not a systematic or recurring one).
  - If the above conditions are not met, corrective action must be taken, and all samples analyzed since the last acceptable CCV must be re-analyzed.

Calculations:

Waters Data System:

1. The Water's Data system automatically calculates sample concentration for each compound of interest. It does this by computing a linear regression for each compound from the data acquired for the calibration standards. This computed calibration curve has the equation:

$$Y = mX + b$$

Where:
- Y = response in height units
- m = slope ("B" on the Water's system)
- X = concentration in ppb (ug/L for waters, ug/kg for soils)
- b = y-intercept ("A" on the Water's system)

Therefore, for sample concentrations the equation would be:

$$X = \frac{(Y-b)}{(m)} \times \text{dilution factor}$$

The dilution factor can be added into the Water's system by entering the correct factor under "Scale Factor" in the multi method (see Water's Operation Manual).

Manual Calculations:

1. Relative Percent Difference (%RPD): This calculation is used to figure the precision of each

compound from the matrix spike/matrix spike duplicate.

$$\%RPD = \frac{(A-B)}{(A+B)/2} \times 100$$

Where: A = Matrix spike concentration (ppb)
B = Matrix spike duplicate concentration (ppb)

2. Percent Recovery (% Rec): This calculation is used to figure the accuracy of each compound from the matrix spike/matrix spike duplicate (MS/MSD). It is also used for the QC check standard recovery and the continuing calibration.

$$\%Rec = \frac{A}{B} \times 100$$

Where: A = The computed concentrations (for the MS/MS and QC check standard), or the current response for the 10 ppb continuing calibration standard.

B = The theoretical concentration expected to be detected (for MS/MSD and the QC check standard), or the initial 10 ppb standard response.

ATTACHMENT I
Preparation of Stock Standards

Stock Standard Solutions: Standards can be prepared in-house from neat reference materials or can be purchased as certified solutions. For in-house, neat standard preparation, the stock standard solutions are prepared as follows:

1. Add about 9 mL of methanol to a 10 mL volumetric flask. Allow the flask to stand, unstoppered until all alcohol wetted surfaces have dried. Tare the flask on an analytical balance.

2. Using a 100 uL syringe, add approximately 100 mg of the reference material. Make sure the drops fall directly into the methanol. Record weight and tare flask.

3. Repeat step 2 for each compound of interest, recording the weight each time.

4. After all compounds have been added, dilute to volume with methanol, stopper, and invert three times to mix.

5. Correction for the recorded weight is required for compounds that are less than 96% pure.

6. Those stock standards which are prepared in-house should be prepared fresh monthly. Commercially purchased mixes are good for 1 month after the ampule has been opened. However, stock standards should be prepared fresh more frequently if the analysis of the QC Check standards indicates that loss or degradation has occurred.

Required Stock Solutions

1. VOC Stock Solution #1 (Purchased):

Compound	Concentration
Benzene	100 ug/mL in Methanol
Bromobenzene	100 ug/mL in Methanol
Brommochloromethane	100 ug/mL in Methanol
Bromodichloromethane	100 ug/mL in Methanol
Bromoform	100 ug/mL in Methanol
Bromomethane	100 ug/mL in Methanol
n-Butylbenzene	100 ug/mL in Methanol
sec-Butylbenzene	100 ug/mL in Methanol
tert-Butylbenzene	100 ug/mL in Methanol

Compound	Concentration
Carbon tetrachloride	100 ug/mL in Methanol
Chlorobenzene	100 ug/mL in Methanol
Chloroethane	100 ug/mL in Methanol
Chloroform	100 ug/mL in Methanol
Chloromethane	100 ug/mL in Methanol
2-Chlorotoluene	100 ug/mL in Methanol
4-Chlorotoluene	100 ug/mL in Methanol
Dibromochloromethane	100 ug/mL in Methanol
1,2-Dibromo-3-chloropropane	100 ug/mL in Methanol
1,2-Dibromoethane	100 ug/mL in Methanol
Dibromomethane	100 ug/mL in Methanol
1,2-Dichlorobenzene	100 ug/mL in Methanol
1,3-Dichlorobenzene	100 ug/mL in Methanol
1,4-Dichlorobenzene	100 ug/mL in Methanol
Dichlorodifluoromethane	100 ug/mL in Methanol
1,1-Dichloroethane	100 ug/mL in Methanol
1,2-Dichloroethane	100 ug/mL in Methanol
1,1-Dichloroethene	100 ug/mL in Methanol
cis-1,2-Dichloroethene	100 ug/mL in Methanol
o-Xylene	100 ug/mL in Methanol
m-Xylene	100 ug/mL in Methanol
p-Xylene	100 ug/mL in Methanol
trans-1,2-Dichloroethene	100 ug/mL in Methanol
1,2-Dichloropropane	100 ug/mL in Methanol
1,3-Dichloropropane	100 ug/mL in Methanol
2,2-Dichloropropane	100 ug/mL in Methanol
1,1-Dichloropropene	100 ug/mL in Methanol
1,3-Dichloropropene	100 ug/mL in Methanol
Ethylbenzene	100 ug/mL in Methanol
Hexachlorobutadiene	100 ug/mL in Methanol
Isopropyl benzene	100 ug/mL in Methanol
p-Isopropyl toluene	100 ug/mL in Methanol
Methylene chloride	100 ug/mL in Methanol
Naphthalene	100 ug/mL in Methanol
n-Propyl benzene	100 ug/mL in Methanol
Styrene	100 ug/mL in Methanol
1,1,1,2-Tetrachloroethane	100 ug/mL in Methanol
1,1,2,2-Tetrachloroethane	100 ug/mL in Methanol
Tetrachloroethene	100 ug/mL in Methanol
Toluene	100 ug/mL in Methanol
1,2,3-Trichlorobenzene	100 ug/mL in Methanol
1,2,4-Trichlorobenzene	100 ug/mL in Methanol
1,1,1-Trichloroethane	100 ug/mL in Methanol
1,1,2-Trichloroethane	100 ug/mL in Methanol

Compound	Concentration
Trichloroethene	100 ug/mL in Methanol
Trichlorofluoromethane	100 ug/mL in Methanol
1,2,3-Trichloropropane	100 ug/mL in Methanol
1,2,4-Trimethylbeznene	100 ug/mL in Methanol
1,3,5-Trimethylbenzene	100 ug/mL in Methanol

2. **VOC Stock Solution #2 (Purchased):**

Compound	Concentration
Vinyl chloride	100 ug/mL in Methanol

3. **VOC Stock Solution #3 (Prepared internally):**

Compound	Concentration
Di-isopropylether	10,000 ug/mL in Methanol

4. **VOC Stock Solution #4 (Purchased):**

Compound	Concentration
2-Chloroethylvinyl ether	1000 ug/mL in Methanol

5. **Ketones and Tetrahydrofuran Stock Solution (Prepared internally):**

Compound	Concentration
Acetone	10,000 ug/mL in Methanol
Tetrahydrofuran (THF)	10,000 ug/mL in Methanol
Methyl ethyl ketone (MEK)	10,000 ug/mL in Methanol
Methyl isobutyl ketone (MIBK)	10,000 ug/mL in Methanol
Methyl tert-butyl ether	10,000 ug/mL in Methanol

6. **Acrolein and Acrylonitrile Stock Solution (Prepared internally):**

Compound	Concentration
Acrolein	10,000 ug/mL in Methanol
Acrylonitrile	10,000 ug/mL in Methanol

7. **tert-Butyl Alcohol Stock Solution (Prepared internally):**

Compound	Concentration
tert-Butyl alcohol	10,000 ug/mL in Methanol

8. **Trichlorotrifluoromethane Stock Solution (Prepared internally):**

Compound	Concentration
Trichlorotrifluoromethane	10,000 ug/mL in Methanol

9. **601/602 Surrogate Stock (Prepared internally):**

Compound	Concentration
a,a,a-Trifluorotoluene	10,000 ug/mL in Methanol
1,4-Dichlorobutane	10,000 ug/mL in Methanol

10. **502.2 Surrogate Stock (Prepared internally):**

Compound	Concentration
Fluorobenzene	10,000 ug/mL in Methanol
2-Chlorobutane	10,000 ug/mL in Methanol

11. **Matrix Spiking Solution (Purchased):**

Compound	Concentration
Benzene	250 ug/mL in Methanol
Toluene	250 ug/mL in Methanol
Chlorobenzene	250 ug/mL in Methanol
Trichloroethene	250 ug/mL in Methanol
1,1-Dichloroethene	250 ug/mL in Methanol

12. **QC Check Standard Stock Solution #1 (Purchased):**

	Compound	Concentration
Mix A:	Benzene	200 ug/mL in Methanol
	Bromobenzene	200 ug/mL in Methanol
	Bromochloromethane	200 ug/mL in Methanol
	Bromodichloromethane	200 ug/mL in Methanol
	Bromoform	200 ug/mL in Methanol
	n-Butylbenzene	200 ug/mL in Methanol
	sec-Butylbenzene	200 ug/mL in Methanol
	tert-Butylbenzene	200 ug/mL in Methanol
	Carbon tetrachloride	200 ug/mL in Methanol
	Chlorobenzene	200 ug/mL in Methanol
	Chloroform	200 ug/mL in Methanol
	2-Chlorotoluene	200 ug/mL in Methanol
	4-Chlorotoluene	200 ug/mL in Methanol

Compound	Concentration
Dibromochloromethane	200 ug/mL in Methanol
1,2-Dibromo-3-chloropropane	200 ug/mL in Methanol
1,2-Dibromoethane	200 ug/mL in Methanol
Dibromomethane	200 ug/mL in Methanol
1,2-Dichlorobenzene	200 ug/mL in Methanol
1,3-Dichlorobenzene	200 ug/mL in Methanol
1,4-Dichlorobenzene	200 ug/mL in Methanol
1,1-Dichloroethane	200 ug/mL in Methanol
1,2-Dichloroethane	200 ug/mL in Methanol
1,1-Dichloroethene	200 ug/mL in Methanol
cis-1,2-Dichloroethene	200 ug/mL in Methanol
trans-1,2-Dichloroethene	200 ug/mL in Methanol
1,2-Dichloropropane	200 ug/mL in Methanol
1,3-Dichloropropane	200 ug/mL in Methanol
2,2-Dichloropropane	200 ug/mL in Methanol
1,1-Dichloropropene	200 ug/mL in Methanol
1,3-Dichloropropene	200 ug/mL in Methanol
Ethylbenzene	200 ug/mL in Methanol
Hexachlorobutadiene	200 ug/mL in Methanol
Isopropyl benzene	200 ug/mL in Methanol
p-Isopropyl toluene	200 ug/mL in Methanol
Methylene chloride	200 ug/mL in Methanol
Naphthalene	200 ug/mL in Methanol
p-Propylbenzene	200 ug/mL in Methanol
Styrene	200 ug/mL in Methanol
1,1,1,2-Tetrachloroethane	200 ug/mL in Methanol
1,1,2,2-Tetrachloroethane	200 ug/mL in Methanol
Tetrachloroethene	200 ug/mL in Methanol
Toluene	200 ug/mL in Methanol
1,2,3-Trichlorobenzene	200 ug/mL in Methanol
1,2,4-Trichlorobenzene	200 ug/mL in Methanol
1,1,1-Trichloroethane	200 ug/mL in Methanol
1,1,2-Trichloroethane	200 ug/mL in Methanol
Trichloroethene	200 ug/mL in Methanol
1,2,3-Trichloropropane	200 ug/mL in Methanol
1,2,4-Trimethylbenzene	200 ug/mL in Methanol
1,3,5-Trimethylbenzene	200 ug/mL in Methanol
o-Xylene	200 ug/mL in Methanol
m-Xylene	200 ug/mL in Methanol
p-Xylene	200 ug/mL in Methanol

	Compound	Concentration
Mix B:	Bromomethane	200 ug/mL in Methanol
	Chloroethane	200 ug/mL in Methanol
	Chloromethane	200 ug/mL in Methanol
	Dichlorodifluoromethane	200 ug/mL in Methanol
	Trichlorofluoromethane	200 ug/mL in Methanol
	Vinyl chloride	200 ug/mL in Methanol

Notes:

1. Internally prepared stock standard concentrations are target levels. Actual concentrations may vary slightly.
2. If additional compounds must be spiked, the QC Check standard(s) can be used for this purpose.

ATTACHMENT II
Preparation of Working Standards

1. Secondary dilutions are prepared in methanol at levels that are appropriate for calibration and the preparation of fortified samples for laboratory blanks.
2. Working Standards are prepared fresh on a weekly basis.

Working Stock Standard

Add 1000 uL of the VOC Stock Solution #1, 1000 uL of the VOC Stock Solution #2, 10 uL of the 601/602 surrogate stock solution, 10 uL of the VOC Stock Solution #3, and 100 uL of the VOC Stock Solution #4 and dilute to 5 mL with methanol. This is equal to a 20 ug/mL working stock standard.

Working Surrogate Solution

For Low Soils and Waters:
For the 601/602 working surrogate solution, add 10 uL of the 601/602 stock surrogate solution and dilute to 10 mL with methanol. This is equal to a 10 ug/mL working surrogate solution.

For the 502.2 working surrogate solution, add 10 uL of the 502.2 stock surrogate solution and dilute to 10 mL with methanol. This is equal to a 10 ug/mL working surrogate solution.

For Medium/High Soils:
Same as for low soils and waters added directly into a 5 mL syringe.

Working Matrix Spike Solution

For Low Soils and Waters:
Add 200 uL of the stock spiking solution (250 ug/mL) and dilution to 5 mL with methanol. This is equal to a 10 ug/mL working spike solution.

For Medium/High Soils:
The Stock spiking stolution (250 ug/mL) is used.

Working QC Check Standard Solution

In a 100 mL volumetric containing approximately 50 mL of reagent water, add 5 uL of each of the stock check standard solutions A and B, and dilute to volume. Take a 5 mL aliquot of this and load onto the ALS. This yields a 10 ug/L check standard.

TABLE 1
COMPOUND LISTS AND REPORTING LIMITS

	Waters (ug/L)	Low Soils[1] (ug/kg)	Medium/High Soils (ug/kg)
601/602 and 8010/8020 Compound List			
Benzene	1.0	5.0	50
Bromochloromethane	1.0	5.0	50
Bromodichloromethane	1.0	5.0	50
Bromoform	1.0	5.0	50
Bromomethane	1.0	10	50
Carbon tetrachloride	1.0	5.0	50
Chlorobenzene	1.0	5.0	50
Chlorodibromomethane	1.0	5.0	50
Chloroethane	2.0	10	50
2-Chloroethylvinyl ether	10	50	100
Chloroform	1.0	5.0	500
Chloromethane	2.0	10	100
1,2-Dibromo-3-chloropropane	3.0	15	150
1,2-Dichlorobenzene	1.0	5.0	50
1,3-Dichlorobenzene	1.0	5.0	50
1,4-Dichlorobenzene	1.0	5.0	50
1,1-Dichloroethane	1.0	5.0	50
1,2-Dichloroethane	1.0	5.0	50
1,1-Dichloroethene	1.0	5.0	50
cis-1,2-Dichloroethene	1.0	5.0	50
trans-1,2-Dichloroethene	1.0	5.0	50
1,2-Dichloropropane	1.0	5.0	50
cis-1,3-Dichloropropene	1.0	5.0	50
trans-1,3-Dichloropropene	1.0	5.0	50
Ethylbenzene	1.0	5.0	50
Methylene chloride	3.0	15	150
1,1,1,2-Tetrachloroethane	1.0	5.0	50
1,1,2,2-Tetrachloroethane	1.0	5.0	50
Tetrachloroethene	1.0	5.0	50
Toluene	1.0	5.0	50
1,1,1-Trichloroethane	1.0	5.0	50
1,1,2-Trichloroethane	1.0	5.0	50
Trichloroethene	1.0	5.0	50
Trichlorofluoromethane	1.0	5.0	50
Vinyl chloride	1.0	5.0	50
o-Xylene	1.0	5.0	50
m+p-Xylene	2.0	10	100

Notes:
1. Illinois LUST program requires reporting limits of 1.0 ug/kg.

TABLE 1
(continued)

	Waters (ug/L)	Low Soils (ug/kg)	Medium/High Soils (ug/kg)
601/602 Non-Target List			
Bromobenzene	1.0	5.0	50
2-Chlorotoluene	1.0	5.0	50
4-Chlorotoluene	1.0	5.0	50
Isopropylbenzene (Cumene)	1.0	5.0	50
Styrene	1.0	5.0	50
1,1,2-Trichlorotrifluoroethane	1.0	5.0	50
BETX List			
Benzene	1.0	5.0	50
Ethylbenzene	1.0	5.0	50
Toluene	1.0	5.0	50
Xylenes	3.0	15	150
BETX/PVOC List			
Benzene	1.0	5.0	50
Ethylbenzene	1.0	5.0	50
Methyl tert-butyl ether	1.0	5.0	50
Toluene	1.0	5.0	50
m+p-Xylene	2.0	10	100
o-Xylene	1.0	5.0	50
1,2,4-Trimethylbenzene	1.0	5.0	50
1,3,5-Trimethylbenzene	1.0	5.0	50
Ketones and THF List			
Acetone	20	100	1000
Methyl ethyl ketone (MEK)	20	100	1000
Methyl isobutyl ketone (MIBK)	10	50	500
Tetrahydrofuran	20	100	1000
Miscellaneous Compounds			
Acrolein	100	500	5000
Acrylonitrile	100	500	5000
tert-Butyl Alcohol	1000	5000	50000
Trichlorotrifluoromethane	2.0	10	100

TABLE 1
(continued)

	Waters (ug/L)	Low Soils (ug/kg)	Medium/High Soils (ug/kg)
8021 List			
Benzene	1.0	5.0	50
Bromobenzene	1.0	5.0	50
Bromochloromethane	1.0	5.0	50
Bromodichloromethane	1.0	5.0	50
Bromoform	1.0	5.0	50
Bromomethane	2.0	10	100
n-Butylbenzene	1.0	5.0	50
sec-Butylbenzene	1.0	5.0	50
tert-Butylbenzene	1.0	5.0	50
Carbon tetrachloride	1.0	5.0	50
Chlorobenzene	1.0	5.0	50
Chloroethane	2.0	10	100
Chloroform	1.0	5.0	50
Chloromethane	2.0	10	100
2-Chlorotoluene	1.0	5.0	50
4-Chlorotoluene	1.0	5.0	50
Dibromochloromethane	1.0	5.0	50
1,2-Dibromo-3-chloropropane	3.0	15	150
1,2-Dibromoethane	2.0	10	100
Dibromomethane	1.0	5.0	50
1,2-Dichlorobenzene	1.0	5.0	50
1,3-Dichlorobenzene	1.0	5.0	50
1,4-Dichlorobenzene	1.0	5.0	50
Dichlorodifluoromethane	2.0	10	100
1,1-Dichloroethane	1.0	5.0	50
1,2-Dichloroethane	1.0	5.0	50
1,1-Dichloroethene	1.0	5.0	50
cis-1,2-Dichloroethene	1.0	5.0	50
trans-1,2-Dichloroethene	1.0	5.0	50
1,2-Dichloropropane	1.0	5.0	50
1,3-Dichloropropane	1.0	5.0	50
2,2-Dichloropropane	1.0	5.0	50
1,1-Dichloropropene	1.0	5.0	50
Ethylbenzene	1.0	5.0	50
Hexachlorobutadiene	1.0	5.0	50
Isopropylbenzene	1.0	5.0	50
p-Isopropyltoluene	1.0	5.0	50
Methylene chloride	3.0	15	150
Naphthalene	1.0	5.0	50
n-Propylbenzene	1.0	5.0	50
Styrene	1.0	5.0	50
1,1,1,2-Tetrachloroethane	1.0	5.0	50
1,1,2,2-Tetrachloroethane	1.0	5.0	50
Tetrachloroethene	1.0	5.0	50

TABLE 1
(continued)

	Waters (ug/L)	Low Soils (ug/kg)	Medium/High Soils (ug/kg)
Toluene	1.0	5.0	50
1,2,3-Trichlorobenzene	1.0	5.0	50
1,2,4-Trichlorobenzene	1.0	5.0	50
1,1,1-Trichloroethane	1.0	5.0	50
1,1,2-Trichloroethane	1.0	5.0	50
Trichloroethene	1.0	5.0	50
Trichlorofluoromethane	1.0	5.0	50
1,2,3-Trichloropropane	1.0	5.0	50
1,2,4-Trimethylbenzene	1.0	5.0	50
1,3,5-Trimethylbenzene	1.0	5.0	50
Vinyl chloride	1.0	5.0	50
o-Xylene	1.0	5.0	50
m-Xylene	1.0	5.0	50
p-Xylene	1.0	5.0	50
cis-1,3-Dichloropropene	1.0	5.0	50
trans-1,3-Dichloropropene	1.0	5.0	50
Di-isopropyl ether	1.0	5.0	50
Methyl tert-butyl ether	1.0	5.0	50

Revision Date

5-22-91

8-03-92

12-14-92

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[VOLSOP-666]

B2

TOTAL ALKALINITY

LABORATORY METHOD STANDARD OPERATING PROCEDURE

Section: Wet Chemistry - Automated Methods	**Section Number** 2	**Revision #** 6	**Effective Date** 06-30-93
SOP Description: Alkalinity	**SOP #** 200	**Document No.** [la-200]	**Page** 1 **of** 6
Reviewed By:	**Approved By:**		

ALKALINITY

Scope and Application
This method is applicable to drinking water, surface water, groundwater and wastewater. This SOP is used in conjunction with the "Lachat AE Autoanalyzer" IOP (IOP#: WCIOP-204).

Method Summary
Colorimetric, Automated, Methyl Orange

Method Reference
"Methods for Chemical Analysis of Water and Wastes", March 1983, Method 310.2.

Lachat Instruments, 1992, Method 10-303-31-1-A.

Reporting Limit
10 mg/L as $CaCO_3$

Optimum Range
10 - 500 mg/L

Sample Handling
Refrigerate at 4°C and analyze within 14 days of collection.

Reagents and Apparatus

1. Milli-Q water
2. Deionized water
3. Sodium hydroxide (0.1M)
4. Sodium carbonate, anhydrous primary standard grade
5. Hydrochloric acid (0.1M)
6. Potassium acid phthalate, primary standard grade
7. Methyl orange indicator, Aldrich
8. Filters: Whatman #1, Whatman #4, and 0.45 micron membrane
9. Lachat Autoanalyzer and associated apparatus
10. Class A volumetric flasks and pipets
11. Disposable 4 mL cups
12. Helium gas

Reagent Preparation (Prepare fresh every 6 months, unless otherwise stated.)

1. **Degassed Milli-Q water:** (2 options)
   a. Boil Milli-Q water vigorously for 5 minutes. Cool and store in cubitainer.
   b. Bubble helium, using the fritted gas dispersion tube, through the Milli-Q water (15 min/20L) Store in cubitainer.

2. **Alkalinity spiking solution (10,000 mg/L as $CaCO_3$):** In a 100 mL volumetric flask, dissolve 1.060g of anhydrous primary standard grade sodium carbonate (Na_2CO_3 - dried at 250°C for 4 hours) in approximately 90 mL of deionized water and dilute to the mark. Refrigerate.

3. **Stock alkalinity standard (1,000 mg/L as $CaCO_3$):** In a 1 liter volumetric flask, dissolve 1.060g of anhydrous primary standard grade sodium carbonate (Na_2CO_3- dried at 250°C for 4 hours) in approximately 900 mL of deionized water and dilute to mark. Refrigerate.

4. **Standards:** (Prepare fresh every month.) Dilute to volume using degassed Milli-Q water. Refrigerate.

Concentration of Standard	Letter Identifier	Volume of Alk. Stock	Dilute to
0 mg/L	A	0	200 mL
20 mg/L	B	4	200 mL
50 mg/L	C	10	200 mL
100 mg/L	D	50	500 mL
250 mg/L	E	50	200 mL
500 mg/L	F	100	200 mL

Note: Computer refers to standards by letter.

5. **Sodium hydroxide (0.1M):** In a 1 liter flask, dissolve 4.0g sodium hydroxide (NaOH) and dilute to the mark with deionized water.

6. **Hydrochloric acid (0.1M):** In a 1 liter flask, dilute 8.3 mL of concentrated HCl in deionized water and dilute to the mark.

7. **KHP buffer (0.025M, pH 3.1):** In a 1 liter flask, dissolve 5.10g of primary standard grade potassium acid phthalate (KHP) ($KHC_8H_4O_4$) in approximately 800 mL of Milli-Q water. Add 70 mL of 0.1M HCl, then carefully, add additional acid to bring pH to between 3.10 and 3.12. **Note:** Use only 0.1M HCl to adjust the pH of buffer. Do **not** back adjust the pH with NaOH. Vacuum filter through a 0.45 micron membrane filter. **Store In Glass. Stable for 1 week.** (Adjust pH and filter before each use.)

8. **Methyl orange reagent:** In a 1 liter volumetric flask, dissolve 0.125g of Aldrich methyl orange indicator in about 700 mL of Milli-Q water and dilute to the mark. Mix well and vacuum filter through a 0.45 micron membrane filter before each use. Store in amber glass! **Note:** The amount of methyl orange indicator used may be adjusted to correct for variances in lots.

Notes

1. Samples must be diluted to obtain concentrations within the optimum working range.

2. Turbidity will interfere. Samples must be filtered prior to analysis. (Use Whatman #1 or #4 filter paper.) Record on the data sheet.

3. Color will interfere, dilute the sample and also spike the dilution to confirm the quality of the result. Record on the data sheet.

Procedure

1. **Instrument Conditions:**
   a. Pump speed: 35
   b. Cycle period: 20 seconds
   c. Inject to start of peak period: 9 seconds
   d. Interference filter: 550 nm
   e. Sample loop: 45 cm
   f. Standards for curve set-up: 0, 20, 50, 100, 250, 500 mg/L.

2. Refer to the "Lachat AE Autoanalyzer" IOP (IOP#: WCIOP-204) for the instrument set up procedure.

3. Analyze samples (including QC samples). The ICV/CCV concentration is 100 mg/L (D).

   To Spike: Into a disposable beaker, add 50 uL of the 10,000 mg/L alkalinity spiking solution to 4 mL of sample. The final spike concentration is 125 mg/L.

4. Refer to the "Lachat AE Autoanalyzer" IOP (IOP#: WCIOP-204) for the instrument shut down procedure.

Quality Control

1. Establish a standard curve with the standards listed above. The derived concentration of each calibration standard must be ± 10% of the true value. The derived value for the blank must be below the method reporting limit.

2. Routine QC Requirements:

Audit	Frequency	Control Limits
ICV/CCV (100 mg/L)	After calibration, every 10 readings, and end of run	90 - 110 %
ICB	1 per run (after ICV)	< Reporting Limit
LCS	1 per run (after ICB)	95% CI (80-120% if no limits are provided)
Duplicate	1 per 10 samples	Calculated quarterly limits
Spike	1 per 10 samples	Calculated quarterly limits

3. CLP Protocol QC Requirements:

Audit	Frequency	Control Limits
ICV/CCV (100 mg/L)	After calibration, every 10 readings, and end of run	90 - 110 %
ICB/CCB	After calibration, every 10 readings, and end of run	< Reporting Limit
LCS	1 per run (after ICB)	95% CI (80-120% if no limits are provided)
Duplicate	1 per 10 samples	Refer to specific Project QAPjP
Spike	1 per 10 samples	Refer to specific Project QAPjP

4. Check standards (ICV/CCV) must be within the acceptable control limits or appropriate action shall be taken and a corrective action report completed.

5. Blanks (ICB/CCB) are to be within the acceptable control limits or appropriate action shall be taken and a corrective action report completed.

6. A Laboratory Control Sample (LCS) is a reference standard prepared by an outside source (usually ERA or EPA). Results must be within acceptable control limits or appropriate action shall be taken and a corrective action report completed.

7. Spike recoveries and duplicate RPDs are to be within acceptable control limits or appropriate action shall be taken and a corrective action report completed. For CLP protocol analysis, associated data shall be flagged "*" if duplicate control limits are exceeded, and "N" if spike control limits are exceeded.

Calculation

1. Calculate with Lachat Quikchem software. Refer to the "Lachat AE Autoanalyzer" IOP (IOP#: WCIOP-204) for further detail.

2. Spike calculation: % Recovery = A-B/C x 100

   where: A = spiked sample result,
   B = sample result, and
   C = spike added (125 mg/L).

3. Duplicate calculation: RPD = |Value1-Value2|/((Value1+Value2)/2) x 100

SOP Revision Dates

1. 08-18-87
2. 09-25-87
3. 09-19-88
4. 02-02-90
5. 05-20-91
6. 06-30-93

[QA ADMIN-LA-200]

B3

CHLORIDE

LABORATORY METHOD STANDARD OPERATING PROCEDURE

Section: Wet Chemistry - Automated Methods	Section Number 2	Revision # 6	Effective Date 06-30-93
SOP Description: Chloride	SOP # 202	Document No. [la-202]	Page 1 of 6
Reviewed By:	Approved By:		

CHLORIDE

Scope and Application
This method is applicable to drinking water, surface water, groundwater and wastewater. This SOP is used in conjunction with the "Lachat AE Autoanalyzer" IOP (IOP#: WCIOP-204).

Method Summary
Colorimetric, Automated, Ferricyanide

Method Reference
"Methods for Chemical Analysis of Water and Wastes", March 1983, Method 325.2.

Lachat Instruments 1992, Method 10-117-07-1-B

Reporting Limit
2 mg/L

Optimum Range
2 - 100 mg/L

Sample Handling
Refrigerate at 4°C and analyze within 28 days of collection.

Reagents and Apparatus

1. Milli-Q water
2. Deionized water
3. Sodium chloride, primary grade
4. Mercuric thiocyanate reagent
5. Ferric nitrate reagent (0.5M)
6. Combined color reagent
7. Filters: Whatman #1 and Whatman #4
8. Lachat Autoanalyzer and associated apparatus
9. Class A volumetric flasks and pipets
10. Disposable 4 mL cups
11. Helium gas

Reagent Preparation (Prepare fresh every 6 months, unless otherwise stated.)

1. **Degassed Milli-Q water:** (2 options)
   a. Boil Milli-Q water vigorously for 5 minutes. Cool and store in cubitainer.
   b. Bubble helium, using the fritted gas dispersion tube, through the Milli-Q water (15 min/20L). Store in cubitainer.

2. **Chloride spiking solution (5000 mg/L):** In a 100 mL volumetric flask, dissolve 0.824g of dried primary grade sodium chloride (NaCl - dried at 140°C overnight) in 50 mL of deionized water. Dilute to the mark and invert to mix. Refrigerate.

3. **Stock chloride standard (1000 mg/L):** Dry 2g of primary grade sodium chloride (NaCl) at 140°C overnight. In a 1 liter volumetric flask, dissolve 1.648g of dried primary grade sodium chloride in 500 mL deionized water. Dilute to the mark and invert to mix. Refrigerate.

4. **Standards:** (Prepare fresh every month). Dilute to volume with deionized water. Refrigerate.

Concentration of Standard	Letter Identifier	Volume of Chloride Stock (mL)	Dilute to
0 mg/L	A	0	200 mL
10 mg/L	B	2	200 mL
20 mg/L	C	4	200 mL
50 mg/L	D	25	500 mL
80 mg/L	E	40	500 mL
100 mg/L	F	20	200 mL

Note: Computer refers to standards by letter. **Final volumes are not the same!**

5. **Stock mercuric thiocyanate reagent:** In a 1 liter volumetric flask, dissolve 4.17g of mercuric thiocyanate ($Hg(SCN)_2$) in one liter of methanol. Invert to mix. Store in amber glass. Refrigerate.

   Caution: Mercury is a very toxic metal. **Wear Gloves!**

6. **Stock ferric nitrate reagent (0.5M):** In a 1 liter volumetric flask, dissolve 202.0g of ferric nitrate ($Fe(NO_3)_3 \cdot 9H_2O$ in approximately 800 mL of deionized water. Add 25 mL of concentrated nitric acid and dilute to one liter. Invert to mix. Refrigerate.

7. **Combined color reagent:** Mix 150 mL of stock mercuric thiocyanate solution with 150 mL of stock ferric nitrate reagent and dilute to 1000 mL with deionized water. Vacuum filter through a 0.45 micron membrane filter. Store at room temperature. **Do not refrigerate.** Commercially available.

Notes

1. Samples must be diluted to obtain concentrations within the optimum working range.

2. Any sample with turbidity must be filtered prior to analysis. (Use Whatman #1 or #4 filter paper.) Record on data sheet.

3. Color is an interference, dilute the sample and also spike the dilution to confirm the quality of the result.

Procedure

1. **Instrument Conditions:**
   a. Pump speed: 35
   b. Cycle period: 30 seconds
   c. Inject to start of peak period: 8 seconds
   d. Interference filter: 480nm
   e. Sample loop: 13 cm
   f. Standards for curve set-up: 0, 10, 20, 50, 80, and 100 mg/L

2. Refer to the "Lachat AE Autoanalyzer" IOP (IOP#: WCIOP-204) for the instrument set up procedure.

3. Analyze samples (including QC samples). The ICV/CCV concentration is 50 mg/L (D).

   To Spike: Into a disposable beaker, add 40 uL of the 5,000 mg/L chloride spiking solution to 4 mL of sample. The final spike concentration is 50 mg/L.

4. Refer to the "Lachat AE Autoanalyzer" IOP (IOP#: WCIOP-204) for the instrument shut down procedure.

Quality Control

1. Establish a standard curve with the standards listed above. Note that the calibration curve is calculated in a "piece-wise" fashion and is not linear. Be sure that calibration points describe a smooth curve. The derived concentration of each calibration standard must be ± 10% of the true value. The derived value for the blank must be below the method reporting limit.

2. Routine QC Requirements:

Audit	Frequency	Control Limits
ICV/CCV (50 mg/L)	After calibration, every 10 readings, and end of run	90 - 110 %
ICB	1 per run (after ICV)	< Reporting Limit
LCS	1 per run (after ICB)	95% CI (80-120% if no limits are provided)
Duplicate	1 per 10 samples	Calculated quarterly limits
Spike	1 per 10 samples	Calculated quarterly limits

3. CLP Protocol QC Requirements:

Audit	Frequency	Control Limits
ICV/CCV (50 mg/L)	After calibration, every 10 readings, and end of run	90 - 110 %
ICB/CCB	After calibration, every 10 readings, and end of run	< Reporting Limit
LCS	1 per run (after ICB)	95% CI (80-120% if no limits are provided)
Duplicate	1 per 10 samples	Refer to specific Project QAPjP
Spike	1 per 10 samples	Refer to specific Project QAPjP

4. Check standards (ICV/CCV) must be within the acceptable control limits or appropriate action shall be taken and a corrective action report completed.

5. Blanks (ICB/CCB) are to be within the acceptable control limits or appropriate action shall be taken and a corrective action report completed.

6. A Laboratory Control Sample (LCS) is a reference standard prepared by an outside source (usually ERA or EPA). Results must be within acceptable control limits or appropriate action shall be taken and a corrective action report completed.

7. Spike recoveries and duplicate RPDs are to be within acceptable control limits or appropriate action shall be taken and a corrective action report completed. For CLP protocol analysis, associated data shall be flagged "*" if duplicate control limits are exceeded, and "N" if spike control limits are exceeded.

Calculation

1. Calculate with Lachat Quikchem software. Refer to the "Lachat AE Autoanalyzer" IOP (IOP#: WCIOP-204) for further detail.

2. Spike calculation: % Recovery = A-B/C x 100

   where: A = spiked sample result,
   B = sample result, and
   C = spike added (50 mg/L).

3. Duplicate calculation: RPD = |Value1-Value2|/((Value1+Value2)/2) x 100

SOP Revision Dates

1. 08-18-87
2. 09-19-88
3. 09-25-88
4. 02-02-90
5. 05-14-91
6. 06-30-93

[QA_ADMIN-LA-202]

B4

SULFATE

LABORATORY METHOD STANDARD OPERATING PROCEDURE

Section: Wet Chemistry - Automated Methods	Section Number 2	Revision # 5	Effective Date 06-30-93
SOP Description: Sulfate	SOP # 216	Document No. [la-216]	Page 1 of 9
Reviewed By:	Approved By:		

SULFATE

Scope and Application
This method is applicable to drinking water, surface water, groundwater and wastewater. This SOP is used in conjunction with the "Lachat AE Autoanalyzer" IOP (IOP#: WCIOP-204).

Method Summary
Colorimetric, Automated, Methylthymol Blue

Method Reference
"Methods for Chemical Analysis of Water and Wastes", March 1983, Method 375.2.

Lachat Instruments 1992, Method 10-116-10-2-B.

Reporting Limit
10 mg/L

Optimum Range
10 - 200 mg/L

Sample Handling
Refrigerate at 4°C and analyze within 28 days of collection.

Reagents and Apparatus
1. Milli-Q water
2. Deionized water

3. Filters: Whatman #1, Whatman #4, and 0.45 micron membrane
4. Lachat Autoanalyzer and associated apparatus
5. Class A volumetric flasks and pipets
6. Disposable 4 mL cups
7. Helium gas
8. Barium chloride
9. Hydrochloric acid
10. Methylthymol blue (Kodak No. 8068)
11. Sodium hydroxide
12. Ammonium chloride
13. Tetrasodium EDTA dihydrate
14. Ammonium hydroxide, conc.
15. Sodium sulfate
16. Ion exchange column and resin

Reagent Preparation (Prepare fresh every 6 months, unless otherwise stated.)

1. **Degassing with helium:** (2 options)
   a. Boil Milli-Q water vigorously for 5 minutes. Cool and store in cubitainer.
   b. Bubble helium, using the fritted gas dispersion tube, through the Milli-Q water (15 min/20L). Store in cubitainer.

2. **Carrier (0.3 ppm SO_4):** In a 1 liter volumetric flask, add 0.3 mL of 1000 ppm stock sulfate solution and dilute to mark with degassed Milli-Q water.

3. **Barium chloride solution (6.24mM):** In a 1 liter volumetric flask, dissolve 1.526g of barium chloride dihydrate ($BaCl_2 \cdot 2H_2O$) in 500 mL of degassed Milli-Q water and dilute to 1 liter.

4. **Hydrochloric acid (1.0N):** In a 100 mL volumetric flask, containing approximately 80 mL of Milli-Q water, add 8.3 mL of concentrated hydrochloric acid and dilute to the mark with Milli-Q water.

5. **Barium - MTB color reagent:** (The purity of the methylthymol blue and the alcohol can be critical. **Use the sources stated below.**)

   In a dry 1000 mL volumetric flask, place 0.2364g* of methylthymol blue (3', 3" bis-N, N-bis carboxymethyl)-amino methylthymolsulfon-ephthalein pentasodium salt

(Kodak No. 8068). Add 50 mL of barium chloride solution ("3" above). The solution may be used to aid in the transfer of the dye. Swirl to dissolve. Add 8.0 mL of the 1.0N HCl solution ("4" above) and mix-solution may turn orange. Add 142 mL deionized water and dilute to 1000 mL with ethanol (Aldrich 24.511.9) Mix. The pH of this solution should be 2.5. Prepare this solution the day before use and store it refrigerated in an amber bottle.

* Strength of reagent varies with lot number. The optimum weight of reagent used must be determined specifically for each new lot of MTB to maintenance consistent method sensitivity. This weight is determined by Lachat and supplied with each batch purchased.

6. **Sodium hydroxide (50% stock solution):** Cautiously dissolve 500g of sodium hydroxide (NaOH) in 600 mL of Milli-Q water. Cool and dilute to 1 liter. Store in plastic bottle. **Caution: The solution will become very hot!**

7. **Sodium hydroxide (0.18N):** In a 1 liter volumetric flask, add 14.4 mL of 50% sodium hydroxide ("6" above) to degassed Milli-Q water, and dilute to the mark.

8. **Buffered EDTA (for cleaning manifold):** In a 1 liter volumetric flask, dissolve 6.75g ammonium chloride (NH_4Cl) in 500 mL deionized water. Add 57 mL concentrated ammonium hydroxide and 40.0g tetrasodium EDTA dihydrate. Dissolve by swirling; dilute to the mark with deionized water.

9. **Sulfate spiking solution (10,000 mg/L):** In a 100 mL volumetric flask, dissolve 1.479g of dried sodium sulfate (Na_2SO_4 - dried at 105°C for 2 hours) in Milli-Q water and dilute to 100 mL.

10. **Sulfate stock (1000 mg/L):** Dry approximately 2g of sodium sulfate (Na_2SO_4) at 105°C for 2 hours. Cool in a desiccator. In a 1 liter volumetric flask, dissolve 1.479 g of the dried sodium sulfate in Milli-Q water and dilute to 1 liter. (1.0 mL = 1.0 mg SO_4).

11. **Working standard:** (Prepare fresh every month.) Dilute to volume using degassed Milli-Q water. Refrigerate.

Concentration of Standard	Letter Identifier	Volume of 1000 mg/L Sulfate Standard	Dilute to
0 mg/L	A	0	200 mL
25 mg/L	B	5.0	200 mL
50 mg/L	C	10	200 mL
100 mg/L	D	50	500 mL
150 mg/L	E	30	200 mL
200 mg/L	F	40	200 mL

Note: Final volumes are not the same! Computer refers to standards by letter.

Notes

1. Samples must be diluted to obtain concentrations within the optimum working range.

2. All coils (including waste coil) must be changed at least once a quarter to prevent build-up in lines.

3. **Interferences:**
   a. The cation exchange column removes multivalent cations. Run a mid-range sulfate standard containing a typical concentration of $CaCO_3$ periodically to check performance. Any decrease in peak height should indicate the need to regenerate or replace the resin. (At 600 ppm $CaCO_3$, the column is good for 80 + injections.)

   b. Samples with pH <2 should be neutralized. High acid concentrations can displace multivalent cations from the column.

   c. Color will interfere. Dilute the sample and also spike the dilution to confirm the quality of the result.

   d. Turbidity - turbid samples may be filtered (use Whatman #1 or #4 filter paper) prior to analysis on Lachat.

   e. Orthophosphate also forms a precipitate with barium at high pH. Check the response of pure orthophosphate standards, if samples are known to be high in phosphate.

4. **Troubleshooting:** (Baseline noise with reagents pumping)
   a. Noise with column in line but good baseline without column:
      - Repack column, **air bubbles** may be causing pulsing.
      - Check flow fit connectors and end fittings on column for blockage or leaks.

   b. Noise with and without column in line:
      - Degas carrier and/or reagents. Fine bubbles cause sharp spikes on baseline.
      - Place a longer piece of manifold tubing on the outlet of the flow cell leading to the waste container. This method requires the use of the screw type flow cell.
      - Replace the pump tubes. The **silicone tube**, used for the color reagent, wears faster than the PVC pump tubes.
      - With water pumping in the lines, check all hydraulic connections for blockages, leaks, etc.

5. **Troubleshooting:** (Baseline drift)
   a. Clean the manifold with the buffered EDTA.
   b. Turn the gain high and use the shortest sample loop possible. This improves the linearity of the calibration curve, prolongs the useful life of the column, and minimizes the build up of $BaSO_4$ on the manifold tubing.

Procedure

1. **Instrument Conditions:**
   a. Pump speed: 35 seconds
   b. Inject to start of peak period: 9 seconds
   c. Cycle period: 50 seconds
   d. Interference filter: 460 nm
   e. Sample loop: 10 cm
   f. Standards for curve set-up: 0, 25, 50, 100, 150, 200 mg/L.

2. Refer to the "Lachat AE Autoanalyzer" IOP (IOP#: WCIOP-204) for the instrument set up procedure.

3. Pump reagents through the lines **before** inserting the column. (Prepare and activate the ion exchange column as described below.) Use a short piece of manifold tubing in place of the column. When all air has passed and the baseline is steady, turn **off** the pump and insert the column. The column should be placed in a vertical position

in place of the column. When all air has passed and the baseline is steady, turn **off** the pump and insert the column. The column should be placed in a vertical position with flow in the top and out the bottom. In this configuration, the column will operate effectively even if the resin packs down more to leave a gap at the top. Resume pumping.

4. **Preparation of Ion Exchange Column:**
   a. Make a slurry of approximately 0.5g of BioRex 70, 50-100 mesh ion exchange resin in Milli-Q water.

   b. Remove one column end from the glass column. Fill the column with water, then aspirate the slurry or allow it to settle by gravity to pack the column. Take care to **avoid trapping air bubbles** in the column and its fittings at this point and all subsequent operations.

   c. After the resin has settled, replace the end fitting. To ensure a good seal, remove any resin particles from the threads of the glass, the column end and the end fittings. To store the column, the ends of the Teflon tubing may be joined with a union.

   d. To test the effectiveness of the column, make up a standard of pure sodium sulfate and compare its peak height to an identical standard with hardness typical of the samples added. If the column is being depleted, the standard with hardness will read lower because the divalent cations are complexing the free MTB. The concentration of the standard should be mid-range. If depletion has occurred, repack the column with fresh resin.

   e. **Regenerating Resin:** Batch regeneration is recommended because the hydrogen form of BioRex 70 can swell considerable more than the sodium form. Collect the used resin in a small beaker or flask. Wash with dilute HCl until the wash tests free of calcium and/or magnesium. This procedure removes the divalent cations by converting the carboxylate exchange group to the protonated form - COOH. Convert the resin back to the sodium form by neutralizing with washes of 0.5M NaOH until the wash has a pH of 9 or greater. Rinse with deionized water for storage or repacking. A column may be used for 3-4 trays (approximately 150 samples) before it needs to be replaced.

SUL

5. Analyze samples (including QC samples). The ICV/CCV concentration is 100 mg/L (D).

   To Spike: Into a disposable beaker, add 30 uL of the 10,000 mg/L sulfate spiking solution to 4 mL of sample. The final spike concentration is 75 mg/L.

6. To shut down, turn off pump and remove the column as follows:
   a. Turn off the pump.
   b. Remove the column.
   c. Join ends of the column with a union.
   d. Replace the column on the manifold with the short teflon tubing piece.
   e. Rinse manifold with Milli-Q water.
   f. Rinse manifold with EDTA cleaning solution.

7. Refer to the "Lachat AE Autoanalyzer" IOP (IOP#: WCIOP-204) for the instrument shut down procedure.

Quality Control

1. Establish a standard curve with the standards listed above. Note that the calibration curve is calculated in a "piece-wise" fashion and is not linear. Be sure that calibration points describe a smooth curve. The derived concentration of each calibration standard must be ± 10% of the true value. The derived value for the blank must be below the method reporting limit.

2. Routine QC Requirements:

Audit	Frequency	Control Limits
ICV/CCV (100 mg/L)	After calibration, every 10 readings, and end of run	90 - 110 %
ICB	1 per run (after ICV)	< Reporting Limit
LCS	1 per run (after ICB)	95% CI (80-120% if no limits are provided)
Duplicate	1 per 10 samples	Calculated quarterly limits
Spike	1 per 10 samples	Calculated quarterly limits

3. CLP Protocol QC Requirements:

Audit	Frequency	Control Limits
ICV/CCV (100 mg/L)	After calibration, every 10 readings, and end of run	90 - 110 %
ICB/CCB	After calibration, every 10 readings, and end of run	< Reporting Limit
LCS	1 per run (after ICB)	95% CI (80-120% if no limits are provided)
Duplicate	1 per 10 samples	Refer to specific Project QAPjP
Spike	1 per 10 samples	Refer to specific Project QAPjP

4. Check standards (ICV/CCV) must be within the acceptable control limits or appropriate action shall be taken and a corrective action report completed.

5. Blanks (ICB/CCB) are to be within the acceptable control limits or appropriate action shall be taken and a corrective action report completed.

6. A Laboratory Control Sample (LCS) is a reference standard prepared by an outside source (usually ERA or EPA). Results must be within acceptable control limits or appropriate action shall be taken and a corrective action report completed.

7. Spike recoveries and duplicate RPDs are to be within acceptable control limits or appropriate action shall be taken and a corrective action report completed. For CLP protocol analysis, associated data shall be flagged "*" if duplicate control limits are exceeded, and "N" if spike control limits are exceeded.

Calculation

1. Calculate with Lachat Quikchem software. Refer to the "Lachat AE Autoanalyzer" IOP (IOP#: WCIOP-204) for further detail.

2. Spike calculation: % Recovery = A-B/C x 100

where: A = spiked sample result,
B = sample result, and
C = spike added (100 mg/L).

3. Duplicate calculation: RPD = |Value1-Value2|/((Value1+Value2)/2) x 100

SOP Revision Dates

1. 08-18-87
2. 09-25-87
3. 02-05-90
4. 05-20-91
5. 06-30-93

[QA_ADMIN-LA-216]

B5

AMMONIA NITROGEN

LABORATORY METHOD STANDARD OPERATING PROCEDURE

Section: Wet Chemistry Manual Methods	Section Number 3	Revision # 3	Effective Date 06-30-93
SOP Description: Nitrogen, Ammonia	SOP # 321	Document No. [wc-321]	Page 1 of 8
Reviewed By:	Approved By:		

AMMONIA NITROGEN

Scope and Application

This method is applicable to the determination of ammonia-nitrogen in drinking water, surface water, groundwater, sludges, soils, and industrial wastes.

Method Summary

Samples are distilled into a solution of boric acid. Ammonia in the distillate is determined colorimetrically by nesslerization.

Method Reference

"Methods for Chemical Analysis of Water and Wastes", March 1983, Method 350.2.

"Standard Methods for the Examination of Water and Wastewater", 17th Edition, 1989, Method 4500-NH_3-B and C.

Reporting Limit

0.10 mg/L for aqueous samples
5.00 mg/kg for soils and sludges

Optimum Range

0.10 - 2.00 mg/L for aqueous samples
5.00 - 100 mg/kg for soils and sludges

Sample Handling

Acidify aqueous samples with concentrated sulfuric acid to pH <2 and refrigerate at 4°C. Refrigerate soils and sludges at 4°C. Analyze within 28 days of sampling.

a. The reservoir should be 2/3 full with Milli-Q water. Add a few boiling chips. Add sulfuric acid to reservoir to bring to a pH <2. Turn on the heater. Set heater control to HIGH. Allow the steam reservoir to heat up. This unit will take about 45 minutes to heat-up. Turn the heater control to about a setting of 8 and bring to boiling. Analysis can begin once boiling begins.

b. Steam out the distillation apparatus with a 10% NaOH solution. Turn on water and continue until 40 mL has been distilled.

3. **Standard Preparation:**

a. Prepare the following series of blanks and standards. Bring pH to 9.5 ± 0.1 using 1N NaOH while stirring slowly.

mL of 10 mg/L Ammonium Chloride Solution	Dilute to	Concentration (mg/L)
0	50 mL	Blank
0.5	50 mL	0.10
1.0	50 mL	0.20
2.0	50 mL	0.40
5.0	50 mL	1.00
10.0	50 mL	2.00

4. **Sample Preparation:**

a. Aqueous samples: Place 50 mL or an aliquot of sample diluted to 50 mL in a 150 mL beaker. Record the volume used. Add 1N NaOH while stirring very slowly until the pH is 9.5 ± 0.1 using a pH meter.

To spike: Place 50 mL sample and 5 mL of the 10 mg/L ammonia standard into a beaker, adjust pH to 9.5 ± 0.1 and continue with procedure. The final spike level is 1.0 mg/L.

b. Non-aqueous samples: Sample must be homogenized prior to analysis to ensure a representative sample aliquot. Place approximately 1.0g in a 150 mL beaker. Record weight used. Add 50 mL Milli-Q water and adjust the pH with 1N NaOH, while stirring slowly, to pH 9.5 ± 0.1 using a pH meter.

To spike: Place 1.0g sample, 5 mL of the 10 mg/L ammonia standard in the beaker. Add 50 mL Milli-Q water, adjust pH, and continue with procedure.

5. Transfer the pH-adjusted sample to a 100 mL Kjeldahl flask. Add 2.5 mL of borate buffer.
6. Add 5 mL of boric acid to a 50 mL Erlenmeyer flask and place flask at the condenser outlet with the **tip of the condenser immersed in the boric acid.**
7. Connect the Kjeldahl flask to the distillation apparatus.
8. Close the stopcock on the condensation chamber. Close the drain stopcock. The steam will now pass through the Kjeldahl flask.
9. Steam distill 30-40 mL at a rate of 4-5 mL/min.
10. Rinse tip of condenser into erlenmeyer flask and remove the erlenmeyer flask.
11. Rinse the tip of the condenser and steam outlet into a waste beaker.
12. Continue distilling remaining samples, blanks and standards. When all samples, blanks and standards are distilled, the colorimetric determination can be performed.
13. **Colorimetric Determination:**
    a. Transfer distilled standards, blanks, and samples to 50 mL mixing cylinders and dilute to 50 mL with Milli-Q water. Mix.
    b. Transfer 25 mL of distillate, or an aliquot diluted to 25 mL, to a mixing cylinder.
    c. Add 1 mL of Nessler reagent and mix by inverting several times.
    d. After 20 minutes, read the absorbance on the spectrophotometer, set at 425nm, using the sipper cell. Zero the spectrophotometer to the reagent blank.

Quality Control

1. Establish a standard curve with the standards listed in the procedure section. Record the absorbance check standard (1.00 mg/L) in the absorbance check book. The absorbances should remain consistent from run to run. If not, necessary troubleshooting must be performed before continuing (check wavelength, spectrometer bulb, solutions, etc.) Record the slope, intercept, and correlation coefficient (R) on the data sheet. The intercept should be <RL and R≥0.995.

2. Summary of QC Requirements (Routine Analysis):

Audit*	Frequency	Control Limits
ICV/CCV (1.00 mg/L)	Initially, every 10 readings, and end of run	90-110%
ICB	1 per run (after ICV)	< Reporting Limit (RL)
LCS	1 per run (after distilled standard)	95%CI (80-120% if no limits are provided)
Duplicate	1 per 10 samples	Calculated quarterly limits
Spike	1 per 10 samples	Calculated quarterly limits

* All QC samples must be carried through the distillation process.

3. Summary of QC Requirements (CLP Protocol):

Audit*	Frequency	Control Limits
ICV/CCV (1.00 mg/L)	Initially, every 10 readings, and end of run	90-110%
ICB/CCB	After ICV, every 10 readings, and end of run	< Reporting Limit (RL)
LCS	1 per run	95%CI (80-120% if no limits are provided)
Duplicate	1 per 10 samples	Refer to specific Project QAPjP

Spike	1 per 10 samples	Refer to specific Project QAPjP

- All QC samples must be carried through the distillation process.

4. Check standards (ICV/CCV) must be within the acceptable control limits or appropriate action shall be taken and a corrective action report completed.

5. Blanks (ICB/CCB) are to be within the acceptable control limits or appropriate action shall be taken and a corrective action report completed.

6. A Laboratory Control Sample (LCS) is a reference standard prepared by an outside source (usually ERA or EPA). Results must be within acceptable control limits or appropriate action shall be taken and a corrective action report completed.

7. Spike recoveries and duplicate RPDs are to be within acceptable control limits or appropriate action shall be taken and a corrective action report completed. For CLP protocol analysis, associated data shall be flagged "*" if duplicate control limits are exceeded, and "N" if spike control limits are exceeded.

8. Aqueous and solid/semi-solid samples are separate matrices. Duplicates and spikes are required for each matrix type.

Calculation

1. Aqueous samples: Calculate the concentration in mg/L using linear regression. Record the slope, intercept, and correlation coefficient (R) on the data sheet. Multiply in any dilution factors performed in the distillation and colorimetric steps to obtain the final concentration in mg/L.

2. Non-aqueous samples: Calculate the concentration in mg/L using linear regression. Record the slope, intercept, and correlation coefficient (R) on the data sheet. Multiply in any dilution factors performed in the colorimetric step. Then multiply the result by 50 and divide by the grams of sample used to obtain the final result in mg/kg.

3. Spike Calculation:

$$\% \text{ recovery} = \frac{(\text{conc of spiked sample} - \text{conc of sample})}{(\text{conc. of spike})(\text{dilution factor})} \times 100$$

SOP Revision Dates

1. 01-06-89
2. 02-02-90
3. 06-30-93

[QA_ADMIN-WC-321]

Reagents and Apparatus

1. Kjeldahl flasks, 100 mL
2. Keeney distillation apparatus
3. Spectrophotometer, set at 425nm with sipper cell
4. Erlenmeyer flasks, 50 mL
5. Sulfuric acid, concentrated
6. Milli-Q water
7. pH meter, 0.1 pH unit sensitivity
8. Volumetric glassware, Class A (pipets and flasks)
9. Top loading balance, 0.01g sensitivity
10. Graduated cylinders, 50 mL
11. Mixing cylinders, 50 mL
12. Ammonium chloride (NH_4Cl)
13. Boric acid (H_3BO_3)
14. Mercuric iodide (HgI_2)
15. Potassium iodide (KI)
16. Sodium hydroxide (NaOH)
17. Sodium tetraborate ($Na_2B_4O_7 \cdot 1OH_2O$)
18. Sodium thiosulfate ($Na_2S_2O_3 \cdot 5H_2O$)
19. Analytical balance, 0.0001g sensitivity
20. 150 mL beaker
21. Stir bars and stir plate

Reagent Preparation - (Prepare fresh every 6 months, unless otherwise stated.)

1. **Ammonium chloride stock solution (1000 mg/L):** In a 1 liter volumetric flask, dissolve 3.819g NH_4Cl in approximately 300 mL Milli-Q water and bring to volume. Preserve with H_2SO_4 to a pH<2. Refrigerate.

2. **Ammonium chloride standard solution (10 mg/L):** Dilute 10.0 mL of the ammonium chloride stock solution to 1 liter with Milli-Q water in a volumetric flask. Preserve with H_2SO_4 to a pH<2. Prepare monthly. Refrigerate.

3. **Boric acid solution:** Dissolve 20.0g H_3BO_3 in Milli-Q water and dilute to 1 liter in a volumetric flask.

4. **Nessler reagent:** Dissolve 100g of mercuric iodide and 70g of potassium iodide in about 200 mL of Mill-Q water. Add this mixture **slowly**, while stirring to a **cooled**

B6

TOTAL SUSPENDED SOLIDS
(TSS)

LABORATORY METHOD STANDARD OPERATING PROCEDURE

Section: Wet Chemistry Manual Methods	Section Number 3	Revision # 4	Effective Date 06-30-93
SOP Description: Solids, Total Suspended	SOP # 337	Document No. [wc-337]	Page 1 of 4
Reviewed By:	Approved By:		

TOTAL SUSPENDED SOLIDS

Scope and Application
This method is applicable to drinking water, surface water, groundwater, domestic and industrial wastewaters.

Method Summary
Gravimetric, dried at 103-105°C

Method Reference
"Methods for Chemical Analysis of Water and Wastes", March 1983, Method 160.2.

"Standard Methods for the Examination of Water and Wastewater", 17th Edition, 1989, Method 2540-D.

Reporting Limit
2 mg/L (using a 500 mL sample volume, see note 2)

Optimum Range
Not applicable.

Sample Handling
Refrigerate at 4°C and analyze within 7 days of sampling.

Reagents and Apparatus

1. Glass fiber filters, Whatman GF/C
2. Gelman filtration funnel and support
3. Suction flask, 1000 mL
4. Aluminum foil weighing dishes
5. Graduated cylinder, 100 mL
6. Drying oven set at 103-105°C
7. Desiccator
8. Analytical balance
9. Deionized water

Reagent Preparation

No reagent preparation required.

Notes

1. **Interferences:** Samples with high dissolved solids may have a positive interference, and the filter should therefore be rinsed well with deionized water under vacuum after the sample has gone through the filter.

2. Select a sample volume that will filter without overloading the filter. If the sample is clear, use a 500 mL volume. Likewise, use a lower volume if TSS is expected to be >10 mg/L.

3. The amount of vacuum applied to filter the sample should not be excessive. Excessive vacuum will cause the filter to break down and lose weight.

Procedure

1. All glassware is to be soap and water washed, tap rinsed, and deionized rinsed prior to analysis.

2. Prepare the glass fiber filter by placing it on the filter support, applying vacuum, and rinsing with three-20 mL portions of deionized water through it.

3. Place the filter in an aluminum weighing dish and dry at 103-105°C for one hour.

4. Place in a desiccator, and cool before weighing (at least 15 minutes). Weigh the aluminum dish containing the prepared filter on the analytical balance and record the

weight. Repeat the drying cycle until a consistent weight (± 0.5 mg) is obtained. Filters may be prepared ahead of time and stored in the desiccator until ready to use.

5. Assemble the filtering apparatus. Begin applying vacuum. Wet the filter with a little deionized water.

6. Shake the sample well and pour an appropriate volume into the apparatus using a graduated cylinder. (Start with 100 mL sample and increase volume until sample has difficulty going through the filter.) The use of pipets is required for volumes of 10 mL and less. Record the volume.

7. With the vacuum still on, rinse the apparatus with 3-10 mL portions of deionized water. Continue the vacuum until filtration is complete.

8. With forceps, carefully remove the filter from the support and place in the **same** aluminum dish.

9. Place in the drying oven and dry at 103-105°C for one hour.

10. Cool in a desiccator for one hour and weigh. Record the weight.

11. Repeat drying cycle until a consistent weight (± 0.05 mg) is obtained. Record the final weight.

Quality Control

1. Summary of QC Requirements (Routine Analysis):

Audit	Frequency	Control Limits
Blank	1 per run	< Reporting Limit
LCS	1 per run (after Blank)	95% CI (80-120% if no limits are provided)
Duplicate	1 per 10 samples	Calculated quarterly limits

2. Summary of QC Requirements (CLP Protocol):

Audit	Frequency	Control Limits
Blank	1 per run	< Reporting Limit
LCS	1 per run (after Blank)	95% CI (80-120% if no limits are provided)
Duplicate	1 per 10 samples	Refer to specific Project QAPjP

3. Blanks are to be within the acceptable control limits or appropriate action shall be taken and a corrective action report completed.

4. A Laboratory Control Sample (LCS) is a reference standard prepared by an outside source (usually ERA or EPA). Results must be within acceptable control limits or appropriate action shall be taken and a corrective action report completed.

5. Duplicate RPDs are to be within acceptable control limits or appropriate action shall be taken and a corrective action report completed. For CLP protocol analysis, associated data shall be flagged "*" if duplicate control limits are exceeded.

Calculations

TSS, mg/L = (A-B)/C x 1000000

where: A = weight of filter, aluminum pan and residue (g)
B = weight of filter and aluminum pan (g)
C = sample volume (mL)

SOP Revision Dates

1. 07-23-86
2. 05-21-87
3. 02-10-90
4. 06-30-93

B7

TOTAL DISSOLVED SOLIDS
(TDS)

WARZYN INC.

LABORATORY METHOD STANDARD OPERATING PROCEDURE

Section: Wet Chemistry Manual Methods	Section Number 3	Revision # 4	Effective Date 06-30-93
SOP Description: Solids, Total Dissolved	SOP # 336	Document No. [wc-336]	Page 1 of 4
Reviewed By:	Approved By:		

TOTAL DISSOLVED SOLIDS

Scope and Application

This method is applicable to drinking water, surface water, groundwater, and domestic and industrial wastewaters.

Method Summary

Gravimetric, dried at 180°C.

Method Reference

"Methods for Chemical Analysis of Water and Wastes", March 1983, Method 160.1.

"Standard Methods for the Examination of Water and Wastewater", 17th Edition, 1989, Method 2540-C.

Reporting Limit

20 mg/L (using a 50 mL sample volume)

Optimum Range

Not applicable.

Sample Handling

Refrigerate at 4°C and analyze sample within 7 days of sampling.

Reagents and Apparatus

1. Glass fiber filters, Whatman GF/C
2. Gelman filtration funnel and support
3. Suction flask, 1000 mL
4. Porcelain evaporating dishes
5. Volumetric pipet, 50 mL
6. Drying oven at 180°C ± 2°C
7. Desiccator
8. Analytical balance
9. Deionized water

Reagent Preparation

No reagent preparation required.

Notes

1. **Interferences:** Samples with high concentrations of bicarbonate, Ca, Mg, Cl, and SO_4 will require prolonged drying, desiccation, and rapid weighing.

2. Total residue should be < 200 mg. Excessive residue (>200 mg) is difficult to dry thoroughly. Use a smaller volume if TDS is suspected to be high; likewise use a larger volume if TDS is suspected low and lower detection limit is required.

3. Groundwater samples which have already been filtered through a 0.45 micron membrane filter do not need to be carried through the filtration step of the procedure.

4. If there are many evaporating dishes to be dried and weighed, it may take up to 3-4 hours before cooling sufficiently to use. Inconsistent weights may be a result of the dishes not being sufficiently cooled.

Procedure

1. All glassware is to be soap and water washed, tap rinsed, and deionized rinsed prior to analysis.

2. **Evaporating Dish Preparation:**
   a. If volatile dissolved solids are also to be analyzed, prepare the evaporating dishes by ashing at 550 ± 50°C for one hour in a muffle furnace.

b. Otherwise, heat the dishes at 180 ± 2°C for one hour. Cool in desiccator. Weigh. Record the weight. The dishes must be cool before being weighed (about one hour). Repeat this cycle until a constant weight is obtained (± 0.5 mg).

3. **Filter Preparation:**
   a. Place the glass fiber filter on the filtration support, place the funnel on top, and wash the filter with three 20 mL portions of deionized water while vacuum is applied. Discard the washings. The filters may be prepared ahead of time. If this is the case, dry them for 1 hour at 103 - 105°C and store in the desiccator until needed.

4. Assemble the filtering apparatus, place a prepared filter on the support and apply vacuum. Rinse filtration set-up with a portion of the sample and discard solution.

5. Pour about 60 mL sample into filtering apparatus. Apply vacuum until all the sample has been filtered.

6. Pipet 50 mL of the filtrate (less, if the sample is expected to have a high dissolved solids content) into a prepared evaporating dish.

7. Evaporate the sample to dryness in the oven at 180 ± 2°C. Cool in a desiccator for at least one hour and weigh. Repeat the drying cycle until a constant weight is obtained (± 0.5 mg).

Quality Control

1. Summary of QC Requirements (Routine Analysis):

Audit	Frequency	Control Limits
Blank	1 per run	< Reporting Limit
LCS	1 per run (after Blank)	95% CI (80-120% if no limits are provided)
Duplicate	1 per 10 samples	Calculated quarterly limits

2. Summary of QC Requirements (CLP Protocol):

Audit	Frequency	Control Limits
Blank	1 per run	< Reporting Limit
LCS	1 per run (after Blank)	95% CI (80-120% if no limits are provided)
Duplicate	1 per 10 samples	Refer to specific Project QAPjP

3. Blanks are to be within the acceptable control limits or appropriate action shall be taken and a corrective action report completed.

4. A Laboratory Control Sample (LCS) is a reference standard prepared by an outside source (usually ERA or EPA). Results must be within acceptable control limits or appropriate action shall be taken and a corrective action report completed.

5. Duplicate RPDs are to be within acceptable control limits or appropriate action shall be taken and a corrective action report completed. For CLP protocol analysis, associated data shall be flagged "*" if duplicate control limits are exceeded.

Calculations

TDS, mg/L = (A-B)/C x 1000000

where: A = weight of dish plus residue (g)
B = weight of dish (g)
C = volume of filtered sample used (mL)

SOP Revision Dates

1. 07-23-86
2. 05-21-87
3. 02-02-90
4. 06-30-93

[QA_ADMIN-WC-336]

B8

TOTAL ORGANIC CARBON (TOC)

TOTAL ORGANIC CARBON

Scope and Application: This method is applicable to surface water, sewage, wastewater and groundwater.

Method: Each standard and sample is sealed in an ampule containing sodium persulfate and phosphoric acid. The ampules are heated for 2 hours at 95°C. The oxidized organic carbon is measured as CO_2 by infra-red detection. Quantitation is determined using an external standard curve.

Reference: EPA 1983: method 415.1, OI Model 524C Total Carbon System Manual

Detection Limit: 1.0 mg/L

Optimum Range: 1.0 - 20 mg/L

Sample Handling: Acidify with concentrated sulfuric acid to pH <2 and refrigerate at 4°C. Analyze within 28 days.

Reagents and Apparatus:

1. OI Model 524C Total Carbon System
2. 10 mL sealed ampules (OI Corp.)
3. Commercial grade O_2 gas
4. Disposable propane gas cylinder
5. Commercial-grade N_2 gas (high purity, dry grade)
6. Eppendorf microliter pipet, 100-1000 uL
7. Oxford macropipet, 0-5 mL
8. Silicone grease
9. Saturated potassium persulfate solution
10. Phosphoric acid solution, 10% v/v
11. Milli-Q water
12. Potassium biphthalate (KHP)
13. Volumetric flasks, 1000 mL and 100 mL
14. Pipets, 1-20 mL assorted
15. EPA quality control sample of known concentration.

Reagent Preparation: (Prepare fresh every six months, unless otherwise stated).

1. **Total Organic Carbon (TOC) Stock Solution, 1000 mg/L:** Dry potassium biphthalate (KHP) at 105°C for 2 hours. Cool in a desiccator. Weigh exactly 2.1254 g dry KHP and add to a 1000 mL volumetric flask containing 2 mL of concentrated H2S04 and Milli-Q water. Mix well and dilute to the mark. Store in refrigerator.

2. **Intermediate TOC Solution (100 ug/mL):** To a 100 mL volumetric flask, pipet 10.0 mL of the 1000 mg/L stock solution. Dilute to the mark with Milli-Q water. **Prepare fresh monthly.**

3. **Standard Carbon Solutions:** (Prepare fresh for each run.)

   - **20 mg/L TOC Standard:** To a 100 mL volumetric flask, pipet 20.0 mL of the 100 mg/L carbon solution, and dilute to the mark with Milli-Q water.

- **10 mg/L TOC Standard:** To a 100 mL volumetric flask, pipet 10.0 mL of the 100 mg/L carbon solution, and dilute to the mark with Milli-Q water.
- **5 mg/L TOC Standard:** To a 100 mL volumetric flask, pipet 5.0 mL of the 100 mg/L carbon solution, and dilute to the mark with Milli-Q water.
- **3 mg/L TOC Standard:** To a 100 mL volumetric flask, pipet 3.0 mL of the 100 mg/L carbon solution, and dilute to the mark with Milli-Q water.
- **1 mg/L TOC Standard:** To a 100 mL volumetric flask, pipet 1.0 mL of the 100 mg/L solution, and dilute to the mark with Milli-Q water.

4. **Potassium persulfate solution ($K_2S_2O_8$), Saturated:** Fill a 1-liter amber glass bottle 3/4 full with Milli-Q water. Add potassium persulfate, approximately 20 g, until no more crystals will dissolve. Store in refrigerator.
5. **Phosphoric acid solution, 10% (v/v):** Slowly add 100 mL of phosphoric acid to 900 mL Milli-Q water on a stirring plate. Mix thoroughly. Store in refrigerator.

Ampule Preparation:

1. Set up run by labeling ampules and placing them in a test tube rack.
2. Snap the ampules open along scoring and replace into rack in run sequence. Discard ampule tips. Keep open ampules covered with aluminum foil to protect from dust and other contaminants.
3. Using an Eppendorf 1000 microliter pipet, carefully pipet 1000 ul of saturated potassium persulfate solution into each ampule.
4. Using a calibrated Oxford Macropipet, carefully pipet 5.0 ml of standard or sample into each ampule. For blanks, pipet 5.0 ml Milli-Q water. For spiked samples pipet 4 ml of sample and 1 ml of the 20 mg/L standard into an ampule.
5. Using Eppendorf microliter pipet, carefully pipet 300 ul of 10% phosphoric acid solution into each ampule.

Ampule Purging and Sealing Procedure:

1. Open oxygen and propane cylinder main valves.
2. Open propane toggle switch and purge propane line for a couple seconds by opening propane adjust valve; then close valve.
3. Light burner while slowly opening propane adjust valve until a flame approximately 2 inches high is obtained.
4. Open oxygen toggle switch. Open oxygen adjust valve slowly until **each** flame has a blue cone in the center.

5. Place 8 ampules in purging rack. Rinse purge tubes with Milli-Q water and insert tubes to the bottom of each ampule.

6. Purge ampules with oxygen for 6 minutes.

7. Place an ampule (purged for 6 minutes) into clamping assembly.

8. Swing microburner into place around ampule tip and seal.

9. Swing microburner back as soon as ampule is sealed.

10. Remove sealed ampule and replace into test tube rack. Open clamping assembly and drop hot ampule tip into a beaker partially filled with water.

11. Rinse purge tube. Place another ampule on purging rack and insert purge tube.

12. Continue with steps 4-8 for remaining ampules.

13. Turn off oxygen and propane main cylinder valves.

14. Place rack of sealed ampules in a 95°C oven for 2 hours. Cool to room temperature.

Instrument Analysis:

1. IR power should be on at all times.
2. Change primary drying tube desiccant (magnesium perchlorate).
3. Open nitrogen cylinder main valve.

4. Analyze ampules.
   - 4.1 Turn integrator power on.
   - 4.2 Place plastic stress adapter and gum rubber seal on neck of first ampule to be read. Silicone grease may be necessary to slide the seal over ampule tip.
   - 4.3 Place ampule in breaking assembly and turn clamping screw until ampule is sealed firmly in place.
   - 4.4 Lower purge tube until it is level with the tip of the ampule.
   - 4.5 Open zero gas valve (nitrogen). Adjust flow to 13 on the flowmeter (200 ml/min). Purge the airspace around ampule tip until IR meter needle returns to zero. For first ampule only, push auto zero button to zero instrument. Clear integrator display.
   - 4.6 Close zero gas valve.
   - 4.7 Raise purge tube above plunger/cutter edges.
   - 4.8 Lower plunger/cutter with a twisting motion, breaking ampule tip.
   - 4.9 Lower purge tube to within 1/8" of ampule bottom.
   - 4.10 Open zero gas valve. Make sure flowrate stabilizes at 13. Purge ampule until integrator stops. Record integrator reading on TOC data sheet.
   - 4.11 Close zero gas valve.
   - 4.12 Remove ampule and discard. Analyze each ampule according to steps 4.2-4.12.

5. Instrument shut-down.

   - Turn off integrator.
   - Close nitrogen cylinder main valve.
   - Clean and lubricate plunger/cutter and barrel assembly.
   - Leave IR power on.

Quality Control:

1. A standard curve consisting of a 20 mg/L, 10 mg/L, 5 mg/L, 3mg/L and 1 mg/L should be analyzed in duplicate. A check standard at 10 mg/L should be analyzed immediately prior to the first sample, every eleventh analysis thereafter and at the end of a run. Recovery of the check standard should be 90% - 110%. If a recovery is outside the Q.C. limits, all samples must be reanalyzed back to the last acceptable check standard.

2. A blank should be analyzed in each run. Results should be less than the detection limit.

3. An EPA quality control sample should be analyzed immediately after the standard curve. Results should be within the 95% confidence limits.

4. A duplicate should be analyzed with each set of 10 samples. If less than 10 samples are analyzed a duplicate is still required. See the latest laboratory Q.C. tables for acceptable limits.

5. A spike should be analyzed with each set of 10 samples. If less than 10 samples are analyzed a spike is still required. See the latest laboratory Q.C. tables for acceptable limits.

6. If any instrument reading is above the range of the standards, the sample must be diluted and repeated. The diluted level must be between 3 mg/L and 20 mg/L or redilute at a level that is within this range and reanalyze.

7. All reagent preparation as well as sample dilutions, and spikes should be documented in a logbook.

Calculations:

1. **Standardization:** Average responses for duplicate curves. Plot concentration vs. integrator response on graph paper.

2. Derive samples concentrations from graph as appropriate. Report results to 1 place past the decimal point from 1 to 99.9 and to 3 significant figures above 100.

Revision Date

9-25-87

3-07-88

11-14-90

8-2-91

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B9

CHEMICAL OXYGEN DEMAND (COD)

LABORATORY METHOD STANDARD OPERATING PROCEDURE

Section: Wet Chemistry Manual Methods	Section Number 3	Revision # 4	Effective Date 06-30-93
SOP Description: Chemical Oxygen Demand	SOP # 305	Document No. [wc-305]	Page 1 of 6
Reviewed By:	Approved By:		

CHEMICAL OXYGEN DEMAND

Scope and Application
This method is applicable to surface water, sewage, wastewater, and groundwater.

Method Summary
Dichromate reflux, Colorimetric

Method Reference
"Methods for Chemical Analysis of Water and Wastes", March 1983, Method 410.4.

"Standard Methods for the Examination of Water and Wastewater", 17th Edition, 1989, Method 5220-D.

Reporting Limit
20 mg/L

Optimum Range
20-700 mg/L

Sample Handling
Preserve with sulfuric acid to a pH <2 and refrigerate at 4°C. Analyze within 28 days.

Reagents and Apparatus
1. Dichromate - mercuric sulfate-sulfuric acid digestion solution
2. Silver sulfate-sulfuric acid catalyst solution
3. COD standard and spiking solutions

4. Block digestor, set at 150°C
5. 16 x 100 mm culture tubes with teflon lined screw caps
6. Oxford pipet, 0-5 mL
7. Spectrophotometer, set at 600 nm wavelength with sipper cell
8. Eppendorf microliter pipetter, 10-100 uL
9. 2 Repipette Dispensers, 1000 mL
10. Deionized water

Reagent Preparation (Prepare fresh every 6 months, unless otherwise noted.)

1. **Digestion solution:** Add 10.2g of dried potassium dichromate ($K_2Cr_2O_7$), 33.3g of mercuric sulfate ($HgSO_4$) and 167 mL of concentrated H_2SO_4 to about 500 mL of deionized water; dilute to 1000 mL in a volumetric flask and stir until dissolved. Store in a dark place.

2. **Silver sulfate-sulfuric acid catalyst solution:** Add 22.0g of silver sulfate (Ag_2SO_4) to a 2.5L bottle of conc. H_2SO_4. Stir to dissolve.

3. **COD stock standard, 1000 mg/L:** Carefully weigh 0.8500g of dried potassium acid phthalate (KHP), dissolve in deionized water and dilute to 1 liter in a volumetric flask. Preserve the stock solution with H_2SO_4 (2 mL conc. H_2SO_4/L of solution). Refrigerate.

4. **Working COD standards:** (Prepare fresh monthly and refrigerate. All working standards should be preserved with H_2SO_4, 0.2 mL conc. H_2SO_4 in 100 mL.)

   a. **700 mg/L standard:** To a 100 mL volumetric flask, add 70 mL of 1000 mg/L stock standard and dilute to the mark with deionized water.

   b. **300 mg/L COD standard:** To a 100 mL volumetric flask, add 30 mL of 1000 mg/L stock standard and dilute to the mark with deionized water.

   c. **100 mg/L COD standard:** To a 200 mL volumetric flask, add 20 mL of 1000 mg/L COD stock standard and dilute to the mark with deionized water.

   d. **50 mg/L COD standard:** To a 100 mL volumetric flask, add 5 mL of 1000 mg/L COD stock standard and dilute to the mark with deionized water.

e. **20 mg/L COD standard:** To a 100 mL volumetric flask, add 2 mL of 1000 mg/L COD stock standard and dilute to the mark with deionized water.

5. **COD Spiking Solution, 10,000 mg/L:** Carefully weigh out 0.8500g of dried potassium acid phthalate (KHP) into a 100 mL volumetric flask. Dissolve in deionized water, and dilute to the mark. Refrigerate. Prepare fresh every 2 months.

Notes

1. If a dark green or turquoise color occurs when sample is added or when the tube is being heated; the sample is over the upper limit of the curve and must be diluted and redigested.

2. **Interference:** Chlorides represent a positive interference. Mercuric sulfate is added to the digestion tubes to complex the chloride. Mercuric sulfate can complex up to 2,000 mg/L chloride before reacting with dichromate in the sample. If chloride exceeds 2,000 mg/L, dilute the sample.

3. **Reagents are corrosive and toxic. Avoid skin contact.**

4. Store all standards in the refrigerator.

5. Store the dichromate solution and prepared tubes in a dark area.

Procedure

1. All glassware is to be soap and water washed, tap rinsed and deionized water rinsed prior to analysis. Rinse digestion tubes and caps with deionized water prior to use. Caps deteriorate over time. Discard caps after each use.

2. Into each tube, pipet exactly 1.5 mL of COD digestion solution, using the repipetter dispenser.

3. Using the repipetter dispenser, pipet down the side into each tube exactly 3.5 mL of the silver sulfate-sulfuric acid solution. Avoid any mixing with the digestion solution. These tubes may be stored, with caps having teflon liners, indefinitely. **Store in the dark!**

4. Prepare a standard curve consisting of the following standards: 0, 20, 50, 100, 300, and 700 mg/L COD. The standards are carried through the digestion step.

5. Using the Oxford 0-5 mL pipet, add 2.5 mL of sample, standard, or blank to the tube. Be careful to avoid air bubbles in the pipet tip and to eject all of the sample. Cap tubes tightly and mix by inverting 10-12 times. If cap leaks while mixing, discard and repeat.

   To Spike: Pipet 2.5 mL of sample into a digestion tube. Add 25 uL of the 10,000 mg/L spiking solution. Mix well. This corresponds to a 100 mg/L final COD spike concentration.

6. Place tubes in a block heater at 150°C for 2 hours. Block heater should be preheated at least 1 hour prior to use.

7. Remove tubes from block heater. Cool to room temperature. Read the absorbance on spectrophotometer, set at 600 nm, using the 1 cm cuvette. Samples can be stored in in the dark and read the next day. Do not shake tubes. Initially zero with the blank standard.

Quality Control

1. Establish a standard curve with the standards listed in the procedure section. Record the absorbance check standard in the absorbance check book. The absorbances should remain consistent from run to run. If not, necessary troubleshooting must be performed before continuing (check wavelength, spectrometer bulb, solutions, etc.) Record the slope, intercept, and correlation coefficient (R) on the data sheet. The intercept should be <RL and R≥0.995.

2. Summary of QC Requirements (Routine Analysis):

Audit	Frequency	Control Limits
ICV/CCV (100 mg/L)	Initially, every 10 readings, and end of run	90-110%
ICB	1 per run (after ICV)	< Reporting Limit (RL)
LCS	After ICB	95% CI (80-120% if no limits are provided)

Duplicate	1 per 10 samples	Calculated quarterly limits
Spike	1 per 10 samples	Calculated quarterly limits

3. Summary of QC Requirements (CLP Protocol):

Audit	Frequency	Control Limits
ICV/CCV (100 mg/L)	Initially, every 10 readings, and end of run	90-110%
ICB/CCB	After ICV, every 10 samples, and end of run	< Reporting Limit (RL)
LCS	After ICB	95% CI (80-120% if no limits are provided)
Duplicate	1 per 10 samples	Refer to specific Project QAPjP
Spike	1 per 10 samples	Refer to specific Project QAPjP

4. Check standards (ICV/CCV) must be within the acceptable control limits or appropriate action shall be taken and a corrective action report completed.

5. Blanks (ICB/CCB) are to be within the acceptable control limits or appropriate action shall be taken and a corrective action report completed.

6. A Laboratory Control Sample (LCS) is a reference standard prepared by an outside source (usually ERA or EPA). Results must be within acceptable control limits or appropriate action shall be taken and a corrective action report completed.

7. Spike recoveries and duplicate RPDs are to be within acceptable control limits or appropriate action shall be taken and a corrective action report completed. For CLP protocol analysis, associated data shall be flagged "*" if duplicate control limits are exceeded, and "N" if spike control limits are exceeded.

Calculation

1. Calculate the concentration in mg/L using linear regression. Record the slope, intercept, and correlation coefficient (R) on the data sheet.

2. Spike Calculation:
   % recovery = (conc of spiked sample - conc of sample)/100 x 100

SOP Revision Dates

1. 07-23-93
2. 02-27-87
3. 02-02-90
4. 06-30-93

[QA_ADMIN-WC-305]

B10

BIOCHEMICAL OXYGEN DEMAND (BOD_5)
AND
YSI DISSOLVED OXYGEN (DO)
METER AND PROBE

LABORATORY METHOD STANDARD OPERATING PROCEDURE

Section: Wet Chemistry Manual Methods	Section Number 3	Revision # 3	Effective Date 06-30-93
SOP Description: Biochemical Oxygen Demand	SOP # 302	Document No. [wc-302]	Page 1 of 9
Reviewed By:	Approved By:		

BIOCHEMICAL OXYGEN DEMAND

Scope and Application

This method is applicable to surface water, sewage, wastewater, and groundwater and measures the oxygen utilized during a specified incubation period, typically 5 days. Carbonaceous BOD can also be determined by this method.

Method Summary

5-day incubation @ 20°C

Method Reference

"Standard Methods for the Examination of Water and Wastewater", 17th Edition, 1989, Methods 5210 and 4500-O.

Reporting Limit

1 mg/L

Sample Handling

Refrigerate at 4°C and set up within 24 hours after sampling.

Reagents and Apparatus

1. 20 L cubitainer filled with Milli-Q water
2. Phosphate buffer solution
3. Magnesium sulfate solution
4. Calcium chloride solution
5. Ferric chloride solution

6. Sodium hydroxide (1N)
7. Sulfuric acid (1N)
8. Volumetric pipettes
9. Magnetic stirrer and stir bars
10. pH meter, calibrated
11. BOD incubator, at 20°C
12. Dissolved oxygen probe and meter, YSI
13. 300 mL BOD bottles with stoppers and plastic caps
14. Milli-Q water
15. Glucose - glutamic acid solution
16. Air pump with plastic tubing
17. Nitrification inhibitor
18. Manganous sulfate solution
19. Alkali-iodide-azide reagent
20. Starch solution
21. Sodium thiosulfate (0.025N)
22. 10% HCl solution
23. Graduated cylinders

Reagent Preparation (Prepare fresh every 6 months, unless otherwise noted.)

1. **Phosphate buffer solution:** Dissolve 8.5g potassium dihydrogen phosphate (KH_2PO_4), 21.75g dipotassium hydrogen phosphate (K_2HPO_4), 33.4g disodium hydrogen phosphate heptahydrate ($Na_2HPO_47H_2O$), and 1.7g ammonium chloride (NH_4Cl), in about 500 mL Milli-Q water and dilute to 1 liter in a volumetric flask. The pH of this buffer should be 7.2 without further adjustment.
2. **Magnesium sulfate solution:** Dissolve 22.5g magnesium sulfate ($MgSO_4 \cdot 7H_20$), in Milli-Q water and dilute to 1 liter in a volumetric flask.
3. **Calcium chloride solution:** Dissolve 27.5g anhydrous calcium chloride ($CaCl_2$), in Milli-Q water and dilute to 1 liter in a volumetric flask.
4. **Ferric chloride solution:** Dissolve 0.25g ferric chloride, ($FeCl_3 \cdot 6H_2O$), in Milli-Q water and dilute to 1 liter in a volumetric flask.

5. **Sodium hydroxide (1N):** Dissolve 40.0g of sodium hydroxide (NaOH) in Milli-Q water and dilute to 1 liter.

6. **Sulfuric acid (1N):** Add 28 mL of conc. H_2SO_4 to about 500 mL of Milli-Q water and dilute to 1 liter.

7. **Glucose-glutamic acid solution:** Dry glucose and glutamic acid at 103°C for 1 hour. Add 150mg glucose and 150mg glutamic acid to Milli-Q water and dilute to 1000 mL in a volumetric flask. Place stir bar in flask and mix until dissolved. A 2% solution (6 mL glucose glutamic acid solution/300 mL dilution water) has a concentration of 198 ± 30.5 mg/L BOD. **Prepare fresh each use!**

8. **Nitrification inhibitor:** Available from Hach Chemicals, #2533.

9. **Manganous sulfate solution:** commercially available.

10. **Alkali-iodide-azide reagent:** Using a 1 liter volumetric flasks, dissolve 5.0g NaN_3 in about 500 mL Milli-Q water. add 500.0g NaOH and 135.0g NaI. Stir to dissolve. Dilute to the mark with Milli-Q water. **Handle with care - TOXIC!**

11. **Starch solution:** Using a 100 mL volumetric flask, dissolve 2.0g starch, 0.2g salicylic acid in 50 mL **hot** MIlli-Q water, and dilute to the mark with Milli-Q water. Cool.

12. **Standard sodium thiosulfate titrant (0.025N):** Using a 1 liter volumetric flask, dissolve 6.205g $Na_2S_2O_3 \cdot 5H_2O$ in about 100 mL Milli-Q water. Add 0.4g NaOH pellets and dilute to the mark with Milli-Q water. Standardize as described in the procedure section.

Notes

1. This test is not applicable to samples containing toxic substances, such as heavy metals.

2. Samples and BOD bottles with sample in them should be kept out of light to prevent formation of dissolved oxygen by algae.

3. Discard any reagent if there is any sign of biological growth in the stock bottle.

4. Add amount recommended of nitrification inhibitor to each sample if a nitrification inhibited BOD (Carbonaceous BOD or CBOD) is requested.
5. Avoid samples that contain residual chlorine by sampling ahead of the chlorination process. If chlorinated samples must be analyzed, consult Standard Methods for the pretreatment procedure.
6. A general rule of thumb to keep in mind - if COD's or TOC's are also required, the ratio of the 3 for the most part are as follows: BOD = TOC x 2; BOD = COD/2. Keep in mind that these may not always be true, but it can assist in determining dilutions needed for the BOD analyses.
7. All samples and standards are seeded. Primary treated sewage obtained from MMSD is used. Approximately 1 liter is collected. Pour off into 100 mL plastic bottles, clearly label, and place in freezer for use at a later date. To use frozen seed: Remove from freezer and thaw. Place on a stir plate and stir while seeding samples and standards.

Procedure

1. Wash with hot soapy water and scrub inside of bottles thoroughly. If bottles are visibly dirty, bottles are to be soaked in dichromate solution. Rinse bottles thoroughly with tap water and deionized water.
2. Clean 20 L cubitainer and tubing with 10% HCl. Rinse 3 times with Milli-Q water, and then fill cubitainer with Milli-Q water.
3. Aerate Milli-Q water for at least 1 hour.
4. Fill (to the neck) two 300 mL BOD bottles with aerated Milli-Q water and determine the DO of the aerated water using the Winkler Titration procedure below.
5. **Winkler Titration:**
   a. To each of the two Milli-Q filled BOD bottles, add 2 mL of manganous sulfate solution followed by 2 mL of alkali-iodide-azide solution. Stopper immediately, making sure no air bubbles are in the bottle.

b. Invert several times to mix. Let floc settle to the half-way mark. Shake and let floc settle again.

c. Remove stoppers and immediately add 2 mL concentrated H_2SO_4 to each bottle. Replace stoppers and invert to mix.

d. At this point, the titration should be completed within 45 minutes.

e. Remove stopper and place the 203 mL plastic BOD flask on top of the BOD bottle and invert to fill the plastic flask. (This measures 203 mL of sample.) Empty the contents of the plastic flask into a 500 mL erlenmeyer flask. Repeat for the other BOD bottle.

f. Record the initial buret reading. Titrate with 0.025N sodium thiosulfate to a pale straw color. Add about 1 mL of the starch solution (sample will turn blue). Continue titrating to the disappearance of blue color. Record the final buret reading. Repeat for the other BOD bottle.

g. Calculate the DO (DO = mL titrated) of each sample. Readings must be within ± 0.05mL of each other, or the titration must be repeated. The average of both titration readings are used for calibration of dissolved oxygen meter.

6. **Calibration of the Dissolved Oxygen Meter:**

a. Place probe in a BOD bottle filled with aerated Milli-Q water and turn the probe on.

b. Switch to RED LINE and adjust with the red line knob until the meter needle aligns with the red mark at the 31°C position.

c. Switch to ZERO and adjust to zero with zero control knob.

e. Switch to TEMPERATURE scale. Record the temperature.

f. Switch to an appropriate mg/L scale range and adjust the CALIBRATE knob, while stirring, until the meter reads the calibration value from the Winkler titration. Leave the probe in the bottle for two minutes to verify calibration stability. Readjust if necessary.

7. **Preparation of Dilution Water:**
   a. The aerated water should be at room temperature before making dilutions of samples. If it is cooler than room temperature, air bubbles will form during incubation, thus biasing the results.

   b. Use a graduated cylinder and add 20 mL of each of the following to the aerated Milli-Q water IN THIS ORDER:

      1. Phosphate buffer solution
      2. Magnesium sulfate solution
      3. Calcium chloride solution
      4. Ferric chloride solution

8. **Sample, Blank, and Standard Preparation:**
   a. Prepare a series of seed dilutions to determine the oxygen depletion of the seed by placing 5, 10, and 15 mL of seed into each of 3 BOD bottles and filling with dilution water.

   b. Prepare the glucose/glutamic acid standard as follows: Pipet 6 mL of this solution into a 300 mL BOD bottle, add 1.0 mL of seed, and fill with dilution water. The concentration should be 198 $\pm$30.5 mg/L as BOD.

   c. Blanks are prepared by filling a BOD bottle with dilution water (no seed).

   d. Allow samples to come to room temperature.

   e. Check the pH of the samples and adjust to pH 6.5-7.5 with 1N H_2SO_4 or 1N NaOH if necessary.

   f. Place 1 mL of seed in each 300 mL BOD bottle.

   g. Shake sample thoroughly. Make 4 serial dilutions per sample into the BOD bottles containing the seed. Use the following chart to determine dilutions.

Approximate BOD Range (mg/L)	Sample Volume* (mL)
0 - 6	300
6 - 18	100
20 - 60	30
60 - 180	10
200 - 600	3
600 - 1,800	10mL of 10x dilution
2,000 - 6,000	3mL of 10x dilution
6,000 - 18,000	10mL of 100x dilution
20,000 - 60,000	3mL of 100x dilution
60,000 - 180,000	10mL of 1000x dilution

* Use the prepared dilution water when making dilutions.

g. Fill each bottle with dilution water.

10. Take initial D.O. reading of all standards, blanks, and samples.

11. Check to make sure no air bubbles are present in the bottles. Remove bubbles by gently tapping the sides of the BOD bottle with the glass stopper. Stopper bottles.

18. Place plastic cap on bottles and place in incubator. Incubate for 5 days at 20°C.

19. Take final D.O. reading after five days. (Calibrate meter prior to use as described above.)

Quality Control

1. Summary of QC Requirements (Routine Analysis):

Audit	Frequency	Control Limits
ICV/CCV (glucose/ glutamic acid standard)	Initially, every 10 BOD bottles, and end of run	198 ± 30.5 mg/L
ICB	1 per run (after ICV)	< Reporting Limit (RL)
LCS	1 per run (after ICB)	95% CI (80-120% if no limits are provided)
Duplicate	1 per 10 samples	Calculated quarterly limits

2. Summary of QC Requirements (CLP Protocol):

Audit	Frequency	Control Limits
ICV/CCV (glucose/ glutamic acid standard)	Initially, every 10 BOD bottles, and end of run	198 ± 30.5 mg/L
ICB/CCB	After ICV, every 10 samples, and end of run	< Reporting Limit (RL)
LCS	1 per run (after ICB)	95% CI (80-120% if no limits are provided)
Duplicate	1 per 10 samples	Refer to specific Project QAPjP

3. Check standards (ICV/CCV) must be within the acceptable control limits or appropriate action shall be taken and a corrective action report completed.

4. Blanks (ICB/CCB) are to be within the acceptable control limits or appropriate action shall be taken and a corrective action report completed.

5. A Laboratory Control Sample (LCS) is a reference standard prepared by an outside source (usually ERA or EPA). Results must be within the acceptable control limits or appropriate action shall be taken and a corrective action report completed.

6. Duplicate RPDs are to be within acceptable control limits or appropriate action shall be taken and a corrective action report completed. For CLP protocol analysis, associated data shall be flagged "*" if duplicate control limits are exceeded.

Calculations

1. Sample concentrations with a DO drop of less than 2 mg/L have "insufficient dissolved oxygen drop" (IDO) and are not reportable unless the maximum sample volume was used (300 mL).

2. Sample concentrations with final DO reading of less than 1 mg/L are considered over range and not reportable.

3. Seed Depletion Calculation (Use the average of the reportable results for SD):

Seed Depletion (SD) = $(B_i - B_f/\text{mL seed used})$

where: B_i = initial DO of seed
B_f = DO of seed after incubation

4. BOD Calculation (Report average of reportable results):

$$\text{BOD (mg/L)} = \frac{(D_i - D_f - SD)(300)(\text{dilution factor})}{\text{mL sample}}$$

where: D_i = initial DO of sample
D_f = DO of sample after incubation
SD = seed depletion

SOP Revision Dates

1. 07-24-86
2. 05-21-87
3. 06-30-93

[QA_ADMIN-WC-302]

B11

NITRATE AND NITRITE NITROGEN

WARZYN INC.

LABORATORY METHOD STANDARD OPERATING PROCEDURE

Section: Wet Chemistry - Automated Methods	Section Number 2	Revision # 4	Effective Date 06/30/93
SOP Description: Nitrate+Nitrite	SOP # 208	Document No. [la-208]	Page 1 of 8
Reviewed By:	Approved By:		

NITRATE+NITRITE NITROGEN

Scope and Application

This method is applicable to drinking water, surface water, groundwater and wastewater. This SOP is used in conjunction with the "Lachat AE Autoanalyzer" IOP (IOP#: WCIOP-204).

Method Summary

Colorimetric, Automated, Cadmium Reduction

Method Reference

"Methods for Chemical Analysis of Water and Wastes", March 1983, Method 353.2.

Lachat Instruments, Method 10-107-04-1-C, September 19, 1991

Reporting Limit

0.02 mg/L

Optimum Range

0.02 - 2.00 mg/L NO_3+NO_2-N

Sample Handling

Preserve with sulfuric acid to pH <2 and refrigerate at 4°C. Analyze within 28 days. Unpreserved samples are to be refrigerated at 4°C, and analyzed within 48 hours of sampling.

Reagents and Apparatus

1. Milli-Q water
2. Deionized water
3. Filters: Whatman #1, Whatman #4, and 0.45 micron membrane
4. Lachat Autoanalyzer and associated apparatus
5. Class A volumetric flasks and pipets
6. Disposable 4 mL cups
7. Helium gas
8. Sulfuric acid
9. Potassium nitrate
10. Sodium hydroxide
11. Ammonium chloride (Baker analyzed, see note 4)
12. Disodium ethylenediamine tetraacetate dihydrate
13. Phosphoric acid
14. Sulfanilamide
15. N-1-Naphthlethylenediamine dihydrochloride (NED)
16. Cadmium powder, coarse granules
17. Acetone
18. Hydrochloric acid
19. Copper sulfate sulution, 2%
20. Polyurethane foam or glass wool

Reagent Preparation (Prepare fresh every 6 months, unless otherwise stated.)

1. **Degassed Milli-Q water:** (2 options)
   a. Boil Milli-Q water vigorously for 5 minutes. Cool and store in a cubitainer, or
   b. Bubble helium, using the fritted gas dispersion tube, through the Milli-Q water (15 min/20L). Store in cubitainer.

2. **Stock nitrate standard (100 mg/L NO_3):** In a 1 liter volumetric flask, dissolve 0.7218g potassium nitrate (KNO_3) in about 600 mL of Milli-Q water. Add 2 mL of concentrated H_2SO_4 as a preservative. Dilute to the mark. Store in a dark glass bottle. Refrigerate.

3. **Working stock nitrate standard (10 mg/L NO_3):** Add 600 mL deionized water to a 1 liter volumetric flask. Add 2.0 mL concentrated H_2SO_4 and pipet 100 mL of the stock nitrate standard. Dilute to the mark with deionzied water. Refrigerate.

4. **Standards:** (Prepare fresh every month.) Preserve with 0.2 mL H_2SO_4. Dilute to volume with deionized water.

Concentration of Standard	Letter Identifier	Volume (mL) of 10 mg/L Working NO_3 Standard	Dilute to
0 mg/L	A	0	100 mL
0.20 mg/L	B	2.0	100 mL
0.50 mg/L	C	5.0	100 mL
1.00 mg/L	D	10	100 mL
2.00 mg/L	E	20	100 mL

Note: Computer refers to standards by letter.

5. **Sodium hydroxide (15M):** To 250 mL of deionized water, **SLOWLY** add 150.0g NaOH. This solution will get very **HOT**! Swirl to dissolve. Store in a plastic bottle.

6. **Ammonium chloride buffer solution:** In a 1 liter volumetric flask, dissolve 85.0g of ammonium chloride (NH_4Cl) and 1.0g of disodium ethylenediamine tetracetate dihydrate (EDTA) in approximately 800 mL deionized water. Adjust the pH to 8.5 with 15M NaOH (approximately 8 mL). Dilute to the mark and filter through a 0.45 um filter.

7. **Sulfanilamide color reagent:** In a 1 liter volumetric flask, add approximately 800 mL of Milli-Q water. Then add 100 mL concentrated phosphoric acid (H_3PO_4). Add 40.0g sulfanilamide and dissolve completely. Dissolve 1.0g N-1-naphthlethylenediamine dihydrochloride (NED) and dilute to one liter. Store in **dark bottle** at 4°C. Stable for **2 months** when refrigerated.

Notes

1. **Interferences:**
   a. Build up of suspended matter in the reduction column will restrict sample flow. Since nitrate-nitrogen is found in a soluble state, the sample must be pre-filtered.

   b. Low results may be obtained for samples that contain high concentrations of iron, copper or other metals. EDTA is added to the samples to eliminate these interferences.

c. Samples that contain large concentrations of oil and grease will coat the surface of the column. This interference is eliminated by pre-extracting the sample with an organic solvent.

2. Samples must be diluted to obtain concentrations within the optimum working range.

3. Sample color will interfere. Reduce interference by diluting the sample. Also spike the diluted sample to confirm the quality of the result. Record on data sheet.

4. ACS grade ammonium chloride has been found occasionally to contain significant nitrate contamination, so an alternative preparation for the ammonium chloride buffer (Reagent #6) is as follows:

   In the hood, add 126 mL concentrated HCl to a 1 liter volumetric flask containing 500 mL degassed Milli-Q water. Mix. Add 95 mL ammonium hydroxide and 1.0 gm disodium EDTA. Dissolve and dilute to the mark. The pH should be 8.5 ± 0.1, adjust pH if necessary.

Procedure

1. **Instrument Conditions:**
   a. Pump speed: 35
   b. Cycle period: 40 seconds
   c. Inject to start of peak period: 15 seconds
   d. Interference filter: 520 nm
   e. Sample loop: 17 cm
   f. Standards for curve set-up: 0, 0.20, 0.50, 1.00, 2.00 mg/L
   g. Column: cadmium reduction

2. Refer to the "Lachat AE Autoanalyzer" IOP (IOP#: WCIOP-204) for the instrument set up procedure.

3. After pumping reagents through the lines, turn control to put column in line, making sure that air bubbles are not introduced into the column. Prepare and activate column as described in following step if necessary.

4. **Column Preparation:** (as necessary)
   a. **Cadmium preparation:** Place 10-20g of coarse cadmium powder (granules) in a 250 mL beaker and wash with 50 mL of acetone, then distilled water, then two 50 mL portions of 1M hydrochloric acid (8 mL concentrated hydrochloric acid plus 92 mL deionized water). Then rinse thoroughly with deionized water. If using cadmium for second time, rinse with 1M hydrochloric acid before beginning process. **CAUTION**: Collect and store all waste cadmium. **Wear Gloves**!

   b. **Copperization:** Prepare a 2% copper sulfate solution (20g $CuSO_4 5H_20$ per liter of deionized water) and add a 100 mL portion to the cadmium prepared in "a" above. Swirl gently for about 5 minutes, then decant the liquid and repeat with a fresh 100 mL portion of 2% copper sulfate. Continue this process until colloidal copper is visible in the supernatant (a red-brown precipitate) and solution remains blue in color. Rinse with deionized water until all colloidal copper is removed from the supernatant. Wash once with ammonium chloride buffer. The cadmium should be black or dark gray. The cadmium granules may be stored in a stoppered bottle in ammonium chloride buffer.

   c. **Packing the column (Wear Gloves!):** Place a small piece of polyurethane foam (or glass wool) loosely in the end of the glass tube. Insert the plugged end of the glass tube into the column end fitting. Cut a length of "0.032" id teflon tubing 3 to 4 inches longer than the column.

      Insert the teflon tube in the end fitting and fill the whole tube with water, holding the flexible tube in a U-shape so that the ends are level. Place the second end fitting on the other end of the teflon tubing. (Placing a small funnel onto the end fitting may aid filling.) Taking care that no air bubbles are introduced, place the copperized cadmium granules in the column. Tap the column gently, every 1-2 cm, to pack the granules. When the column is packed to within about 5 mm of the end of the glass column, insert another foam plug, then the column end fitting. Store the column with the ends connected with a length of teflon tubing, as air pockets or having the column dry out will necessitate repacking. If air remains in the column, connect the column to the manifold and turn the pump on maximum. Tap column firmly until all air is removed.

d. **Column activation:** The column must be activated before use or it will not reduce nitrate. This may be accomplished by pumping the 10 mg/L nitrate standard through the sample line. When the solution is injected, a brilliant pink color will be visible in the coil. The cadmium column efficiency should be above 80%, if less, the column must be repacked.

To check column efficiency, standardize with nitrite standards and then analyze all nitrate standards. The recovery of the nitrate standard should be consistent at all standard concentrations.

5. Analyze samples (including QC samples). The ICV/CCV concentration is 1.00 mg/L (D).

To Spike: Into a disposable beaker, add 20 uL of the 100 mg/L nitrate stock solution to 4 mL of sample. The final spike concentration is 0.50 mg/L.

6. After use, turn off the pump and turn the control to take the column out of line.

7. Refer to the "Lachat AE Autoanalyzer" IOP (IOP#: WCIOP-204) for the instrument shut down procedure.

Quality Control

1. Establish a standard curve with the standards listed above. The derived concentration of each calibration standard must be ± 10% of the true value. The derived value for the blank must be below the method reporting limit.

2. Routine QC Requirements:

Audit	Frequency	Control Limits
ICV/CCV (1.00 mg/L)	After calibration, every 10 readings, and end of run	90 - 110 %
ICB	1 per run (after ICV)	< Reporting Limit
LCS	1 per run (after ICB)	95% CI (80-120% if no limits are provided)
Duplicate	1 per 10 samples	Calculated quarterly limits
Spike	1 per 10 samples	Calculated quarterly limits

3. CLP Protocol QC Requirements:

Audit	Frequency	Control Limits
ICV/CCV (1.00 mg/L)	After calibration, every 10 readings, and end of run	90 - 110 %
ICB/CCB	After calibration, every 10 readings, and end of run	< Reporting Limit
LCS	1 per run (after ICB)	95% CI (80-120% if no limits are provided)
Duplicate	1 per 10 samples	Refer to specific Project QAPjP
Spike	1 per 10 samples	Refer to specific Project QAPjP

4. Check standards (ICV/CCV) must be within the acceptable control limits or appropriate action shall be taken and a corrective action report completed.

5. Blanks (ICB/CCB) are to be within the acceptable control limits or appropriate action shall be taken and a corrective action report completed.

6. A Laboratory Control Sample (LCS) is a reference standard prepared by an outside source (usually ERA or EPA). Results must be within acceptable control limits or appropriate action shall be taken and a corrective action report completed.

7. Spike recoveries and duplicate RPDs are to be within acceptable control limits or appropriate action shall be taken and a corrective action report completed. For CLP protocol analysis, associated data shall be flagged "*" if duplicate control limits are exceeded, and "N" if spike control limits are exceeded.

Calculation

1. Calculate with Lachat Quikchem software. Refer to the "Lachat AE Autoanalyzer" IOP (IOP#: WCIOP-204) for further detail.

2. Spike calculation: % Recovery = A-B/C x 100

   where: A = spiked sample result,
   B = sample result, and
   C = spike added (0.50 mg/L).

3. Duplicate calculation: RPD = |Value1-Value2|/((Value1+Value2)/2) x 100

4. If only nitrate is requested, nitrite must be analyzed, then subtracted from the nitrate + nitrite value.

SOP Revision Dates

1. 08-18-87
2. 02-02-90
3. 05-30-91
4. 06-30-93

[QA_ADMIN-LA-208]

C

C

FIELD MEASUREMENT STANDARD OPERATING PROCEDURES (SOPs)

C1

pH

pH

Scope and Application: This method is applicable to surface water, wastewater and groundwater.

Method: Potentiometric

Reference: EPA 1983, p. 150.1

Sensitivity: 0.01 pH unit

Optimum Range: pH 1.00 to 12.00

Sample Handling: Determine on-site, if possible, otherwise within 24 hours.

Reagents and Apparatus:

1. pH meter
2. Combination pH electrode.
3. Magnetic stirrer and stir bars (for lab use).
4. Beakers or plastic cups.
5. pH buffer solutions, pH 4.00, 7.00, and 10.0.
6. Deionized water

Notes:

1. The pH test is temperature dependent. Therefore, temperatures of buffers and samples should be within 2°C of each other. For refrigerated or cool samples, use refrigerated buffers to calibrate meter.
2. Interferences in pH measurements occur with presence of weak organic and inorganic salts, and oil and grease. If oil and grease are visible, note on data sheet. Clean electrode with soap and water, followed by 10% HCl and deionized water. Then recalibrate meter before analysis of next sample.
3. Electrode should be stored in pH 7.00 buffer, or electrode storage solution.
4. **Before leaving laboratory for field work:**
   a. Check batteries.
   b. Do quick calibration at pH 7.00 and 4.00 to check electrode response and batteries.
   c. Obtain fresh pH buffer solutions.
5. **Following field measurements:**
   a. Report any problems with meter or electrode.

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b. Clean meter and meter case.

c. Make sure electrode is stored in pH 7.00 buffer.

Calibration:

1. Place combination electrode in fresh pH 7.00 buffer solution.
2. After allowing meter to stabilize, turn calibration dial until reading of 7.00 is obtained.
3. Rinse electrode with deionized water and place in pH 4.00 buffer solution.
4. Wait for reading to stabilize and then turn slope adjustment dial until reading of 4.00 is obtained.
5. Rinse electrode with deionized water and place in pH 7.00 buffer. If meter reading is not 7.00 + 0.05, follow Steps 2-5 again.
6. Rinse electrode with deionized water and place in pH 10.00 buffer. Reading must be in the range of 9.90-10.10 or calibration must be repeated.

Procedure:

1. All glassware is to be soap and water washed, tap rinsed and deionized water rinsed prior to analyses.
2. Calibrate meter using calibration procedure.
3. Pour the sample into clean beaker or plastic cup.
4. Place stir bar in beaker and put on magnetic stirrer (low speed) for lab measurement of pH. Swirl cup gently for field measurement of pH.
5. Check temperature of sample. It should be $\pm$ 2°C of the buffer solutions.
6. Rinse electrode with deionized water.
7. Immerse electrode in sample. The white KCl junction on side or bottom of electrode must be fully immersed in solution. Allow sufficient time for reading to stabilize. Record pH. Rinse electrode with deionized water.
8. Check calibration with pH 7.00 buffer solution initially and after every 10 samples and at the end of the analytical run and record on data sheet. Buffer solution should read $\pm$0.05 of true value.

Quality Control:

1. Duplicate 1 out of 10 samples. If less than 10 samples are analyzed, a duplicate is still required. Duplicates should be within acceptable ranges. Average the results.

	Revision Dates
	7-22-86
	6-28-90
Manager, Operations	1-8-92
Supervisor, Quality Assurance	
Kim Johnson Director, Analytical Services	

C2

CONDUCTIVITY YSI METER

CONDUCTIVITY YSI METER

Scope and Application: This method is applicable to surface water, wastewater and groundwater.

Method: Specific Conductance (Electrical Conductivity), umhos/cm @ 25°C

Reference: EPA 1983, Method 120.1

Detection Limit: 10 umhos/cm @ 25°C

Sample Handling: Determine on-site or within 28 days

Reagents and Apparatus:

1. Conductivity meter, YSI 33 SCT
2. Deionized water
3. conductivity standard, 1000 umhos/cm @ 25°C. Commercially Available

Notes:

1. All conductivity readings must be corrected to 25°C.

Procedure:

1. All glassware is to be soap and water washed, tap rinsed and deionized rinsed prior to analysis.

2. With mode switch of the meter in the OFF position. Check the zero setting. If not at zero, use meter adjusting screw to zero (on front of the meter).

3. Plug probe into jack located on side of meter.

4. Turn mode switch to red line, and turn red line knob until needle aligns with red line on dial. Change batteries if meter cannot be aligned.

5. Analyze the conductivity standard. If the result is within 90-110% of the standard value, analyze samples. A standard should be analyzed after every 10 samples and at the end of the analytical run. Table 1 lists non-temperature compensated values for both of the standards. Use this table as a reference when a calculator is not available to perform temperature correction on the standards.

6. Totally immerse and suspend the probe in the water sample. Do not allow probe to touch the sides of the sample container.

7. Turn mode switch to the appropriate conductivity scale (X100, X10, or X1). Use the scale that produces a mid-range output on the meter.

8. Wait for needle to stabilize (about 15 seconds) and record conductivity. Multiply the conductivity reading by the scale setting.

9. While gently agitating the probe, take sample temperature (°C) to nearest 0.5°C and record.

10. Rinse probe with deionized water.

11. Record specific conductivity and temperature.

12. Store probe in D.I. water between uses whenever possible.

13. Turn mode switch to the OFF position when finished.

Quality Control:

1. A quality control calibration standard of 1000 umhos/cm is to be analyzed, initially and after every 10 samples. If less than 10 samples are analyzed, a calibration standard is still required. The last sample analyzed in the run is to be the calibration standard. These standards must be within 90-110% of the standard value or the samples run after the last acceptable check standard are to be reanalyzed.

2. Duplicate a minimum of 1 out of 10 samples. If less than 10 samples are analyzed, a duplicate is still required. Duplicates are to be averaged. Duplicate values are to be within acceptable ranges.

Calculation:

1. **Calculate specific conductivity at 25°C using following formula:**

$$G_{25} = \frac{G_T}{[1 + 0.02\,(T-25)]}$$

G_{25} = Specific conductivity at 25°C, umhos/cm

T = Temperature of sample, °C

G_T = Conductivity of sample at temperature T, umhos/cm

Table 1
NON-TEMPERATURE COMPENSATED CONDUCTIVITY STANDARD READINGS FOR 1,000 AND 10,000 umhos/cm STANDARDS

Conductivity Standard 1,000 umhos/cm			Conductivity Standard 10,000 umhos/cm		
Conductivity Standard Should Read	at	Degrees C°	Conductivity Standard Should Read	at	Degrees C°
500		0	5000		0
520		1	5200		1
540		2	5400		2
560		3	5600		3
580		4	5800		4
600		5	6000		5
620		6	6200		6
640		7	6400		7
660		8	6600		8
680		9	6800		9
700		10	7000		10
720		11	7200		11
740		12	7400		12
760		13	7600		13
780		14	7800		14
800		15	8000		15
820		16	8200		16
840		17	8400		17
860		18	8600		18
880		19	8800		19
900		20	9000		20
920		21	9200		21
940		22	9400		22
960		23	9600		23
980		24	9800		24
1000		25	10,000		25
1020		26	10,200		26
1040		27	10,400		27
1060		28	10,600		28
1080		29	10,800		29
1100		30	11,000		30
1120		31	11,200		31
1140		32	11,400		32
1160		33	11,600		33
1180		34	11,800		34
1200		35	12,000		35

	Revision Date
	9-06-86
	4-27-87
	2-17-88
	2-09-90
Manager, Operations	1-4-92
Supervisor, Quality Assurance	
Director, Analytical Services	

C3

TEMPERATURE

Effective Date: 3-13-92

FIELD TEMPERATURE

Scope and Application: This method is applicable to surface water, wastewater, and groundwater.

Method: Thermometric

Reference: EPA 1983, Method 170.1

Reagents and Apparatus:

1. Thermometer, mercury-filled, or temperature probe, °C
2. Plastic bottles, 250 mL
3. Deionized water
4. Kimwipes

Notes:

1. The thermometer or temperature probe must be calibrated annually against an ASTM certified thermometer.
2. It is recommended the thermometer or temperature probe have a range of -10 to 100°C with 0.5° sensitivity.

Procedure:

1. Pour approximately 100-150 mL sample into a plastic bottle.
2. Place thermometer or temperature probe in sample and swirl gently for 30-60 seconds.
3. Record the temperature to the nearest 0.5 degree.
4. Rinse thermometer with deionized water and blot dry with a kimwipe.
5. Continue as above with next sample.

Quality Control:

1. Duplicate minimum of 1 out of 10 samples. If less than 10 samples are analyzed, a duplicate is still required. Duplicates are to be averaged. Duplicate values are to be within acceptable ranges.

C4

GASTECHTOR GAS SURVEYOR FOR OXYGEN AND COMBUSTIBLE GASES (METHANE)

INSTRUMENT OPERATION PROCEDURE

GASTECHTOR Model 1939 Two-Gas Meter

For the Detection of Oxygen and
Explosive/Combustible Gases

January, 1991

SECTION 1
SCOPE AND APPLICATION

The GASTECHTOR (GASTECH) Model 1939 OX Gas Surveyor is a battery-powered portable instrument that can detect and indicate concentrations of combustible gases and oxygen in air. Combustible gas is measured in two ranges: 0 to 100% Lower Explosive Limit (LEL) and 0 to 100% by volume. Oxygen is measured over a range of 0 to 25% by volume. The instrument is equipped with a meter and audible alarms that sound whenever gases or oxygen concentrations reach preset levels.

REFERENCE

GASTECH Gas Surveyor Model 1939 OX Instruction Manual

APPARATUS

- GASTECH Model 1939 OX
- Battery charger
- Probe, 10 inch long, 1/4 inch OD plastic tube and dust filter
- Hose, 6 foot flexible nylon-jacketed polyethylene tube
- Calibration kit consisting of cylinders of methane, 2 to 5% LEL and 50 to 100% by volume concentrations

SECTION 2
DESCRIPTION

2.1 METER

Instrument readings are displayed on a meter, visible through a window on the top face of the instrument case. A mark on the scale, BATT CK, represents the minimum permissible battery voltage, as an indication of the state of the charge of the battery. The meter face has three scales.

2.1.1 The top scale is graduated from 0 to 25% O_2 for indicating concentration of oxygen. A calibration CAL mark indicates 21% O_2.

2.1.2 On the middle scale the meter reads gas concentration as 0 to 100% LEL. 100% LEL represents the minimum concentration of vapor in air that will ignite or explode when an ignition source is present.

2.1.3 The bottom non-linear scale reads 0 to 100% gas (%GAS) for indicating high concentrations of combustible gas.

2.2 CONTROLS

The six controls that are used in normal operation of the instrument are arranged on the left side of the instrument as viewed from the rear, and on the top. These controls are recessed to minimize possibility of accidental operation.

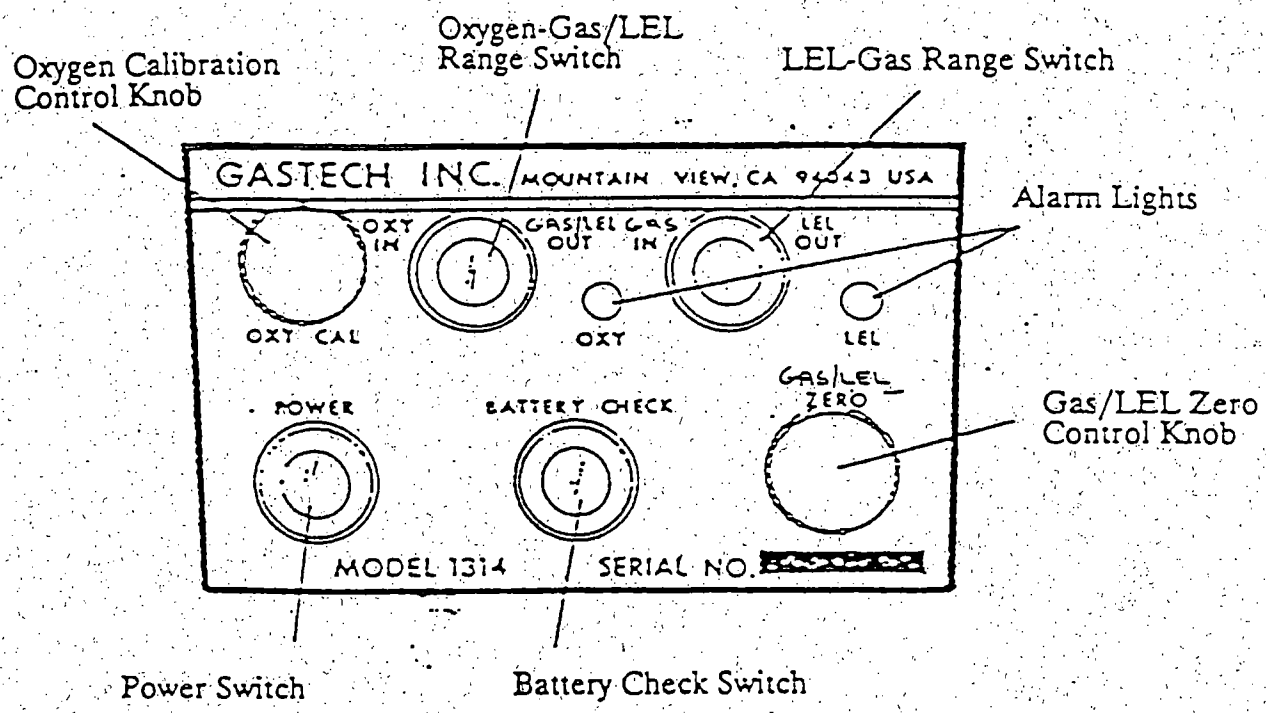

FIGURE 1

2.2.1 Power switch

An alternate-action push button switch which energizes the circuit when pressed. An orange indicator dot is exposed when the switch is in the ON position, serving as a mechanical pilot light.

2.2.2 Battery Check

A momentary push button switch, when pressed connects the meter as a voltmeter for battery condition check.

2.2.3 Gas/LEL Zero

A potentiometer which is used to adjust circuits to read zero in the absence of natural gas. The potentiometer shaft is fitted with a control knob for convenience in adjustment.

2.2.4 OXY/Gas-LEL Switch

An alternate-action push button switch used to select the combustible gas or oxygen range. In the OUT position, the instrument is in the GAS or LEL range. When IN, as indicated by the colored indicator dot, the circuit is in the OXY range.

2.2.5 Gas/LEL Switch

An alternate-action push button switch which selects the operating range for detection of combustible gas. In the OUT position, the instrument measures in the 0 to 100% LEL range. In the IN position, the instrument measures the 0 to 100% GAS range. A colored indicator dot shows when the switch is IN.

2.2.6 OXY Cal

A potentiometer which is used to adjust the circuit to read 21% when the detector is surrounded by normal air. The potentiometer shaft is fitted with a control knob for convenient adjustment.

2.3 INDICATORS

Alarm lights, red and amber, illuminate when the corresponding section of the instrument is in an alarm condition. The red light blinks in an on-off pattern of equal length pulses, to show that the instrument is in the combustible gas (LEL or GAS) alarm condition. Amber light blinks in a short-long pattern to show an abnormal oxygen condition.

2.4 BUZZER

A solid-state electronic buzzer is mounted at the rear interior of the instrument. The buzzer gives a pulsed tone on detection of gas, and a continuous tone in case of a malfunction, either low battery voltage or a down-scale drift of the meter. The audible tone pulses correspond to the visual pulses of the corresponding alarm light except in the case of excess O_2 which gives a steady tone.

TABLE 1
ALARM SETTINGS

% Oxygen:	<19.5% and >25%
% LEL:	>20% LEL
% Gas:	>20% Gas

SECTION 3
PRINCIPLE OF OPERATION

3.1 LEL RANGE

This range gives readings in terms of percent of the lower explosive limit (LEL), with 100% corresponding to the LEL of the specified gas.

Detection in this range is by catalytic oxidation on a platinum element, with the heat of oxidation producing a temperature rise and hence a resistance rise of the element. This change is approximately linear up to the LEL provided there is sufficient oxygen in the sample to support combustion.

3.2 OXYGEN SENSOR

The oxygen-sensing detector is an electrochemical cell in which electrodes are immersed in an alkaline electrolyte, and covered by a permeable fluorocarbon membrane. Oxygen from the surrounding atmosphere diffuses through the membrane and enters the electrolyte, causing an electrochemical reaction. The current generated by this reaction is amplified and used to drive the meter and the alarm circuit.

3.3 100 % GAS RANGE

This range gives readings in terms of gross concentration of the specified combustible gas. It uses a thermal conductivity filament, initially heated by the battery current to a point where it assumes a definite temperature and resistance. The temperature and resistance corresponds to the atmosphere, if the instrument is initially set-up in fresh air. Then when gas is sampled, the greater cooling ability of the gas (relative to air) causes the filament to become cooler and assume a lower electrical resistance. The resistance change produces a meter reading which can be calibrated in units of percent gas by volume.

The thermal conductivity phenomenon is continuous from 0 to 100%, so any concentration can be read without limitation. It is a relatively insensitive method, so is used only for the higher concentrations above the LEL. It is not dependent on combustion, hence is applicable regardless of the oxygen content of the sample.

SECTION 4
OPERATION

4.1 Attach the hose to the instrument by means of the threaded swivel fitting.

4.2 Put the OXY/GAS-LEL switch in the GAS-LEL (out) position, and GAS/LEL switch in the GAS (in) position, with its colored indicator showing.

4.3 Press the POWER switch to turn the instrument on, the orange indicator dot should be showing. The meter will normally rise up scale and a pulsing or steady alarm signal may sound. The alarm will stop when the instrument is warmed-up. The audible hum of the pump will be noticed.

4.4 Press the BATTERY CHECK button and note the meter reading. If the reading is close to or below the BATT CHECK mark on the meter, charge the batteries.

4.5 Allow the unit to warm up until the meter stabilizes (about a minute). Then, with the hose inlet in a contaminant-free location, turn GAS ZERO control knob to bring the meter to "0" indication.

4.6 The instrument is now ready to use in detecting high concentrations of the specified gas up to 100% by volume. The meter will indicate the concentration on the lower non-linear scale.

If the concentration indicated on the meter rises above the alarm point (set at 20%) the red ALARM light will blink and the buzzer will sound, both in a pulsing mode.

4.7 For readings in the LEL range, change the GAS/LEL switch to the LEL (out) position. The alarm will sound and the meter needle will move up-scale briefly. Wait about five minutes for the LEL sensor to stabilize. Move the probe about and observe meter indicators on the middle (% LEL) scale. The alarm point for this scale is set for 20% LEL.

4.8 Put the OXY/GAS-LEL switch in the OXY (in) position with the colored indicator showing. Turn the OXY CAL potentiometer to bring the meter to the oxygen CAL mark (21%). As a quick check gently breathe into the hose inlet and allow the instrument to sample air. The reading should come down to about 16% and the alarm should sound at 19.5%. Allow the meter to return to 21%, then put the switch back in the GAS-LEL (out) position.

4.9 Atmospheres containing more than the normal 21% oxygen will produce an increased oxygen reading. If the instrument is provided with a high oxygen alarm, then it will sound in a steady tone and the amber light will blink when readings reach or exceed 25%.

4.10 The instrument continuously tests for oxygen, and a pulsed audible alarm and blinking amber light will occur when oxygen drops to 19.5% or lower. It is not necessary to use the instrument with the switch in the OXY position unless oxygen meter readings are desired. If both abnormal conditions exist simultaneously (combustible gas and oxygen alarm), both lights will blink in their normal pattern, but the buzzer will sound continuously. A steady tone sounds when the oxygen reading exceeds 25%. This characteristic is provided to warn against the increased fire hazard due to excess oxygen. It also serves as a warning in case of oxygen cell failure in the high-output mode, which can occur occasionally. It further precludes accidental or intentional incorrect adjustment of the oxygen calibration control to an abnormally high level above 25%.

SECTION 5
CALIBRATION AND ADJUSTMENT

5.1 LEL CALIBRATION

5.1.1 Turn the instrument on and allow it to warm up and stabilize, preferably for five minutes. Be sure the batteries are charged sufficiently to read above the check mark.

5.1.2 Place the GAS/LEL switch in the LEL (out) position and the OXY/GAS-LEL switch in the GAS-LEL (out) position.

5.1.3 Open the instrument case by loosening the captive screw at the front of the unit. Lift the upper half of the case slightly, moving it 1/4 inch to the to rear to disengage the rear clamp; then separate the two halves. Locate the COARSE ZERO potentiometer on the underside of the circuit board (see Figure 2).

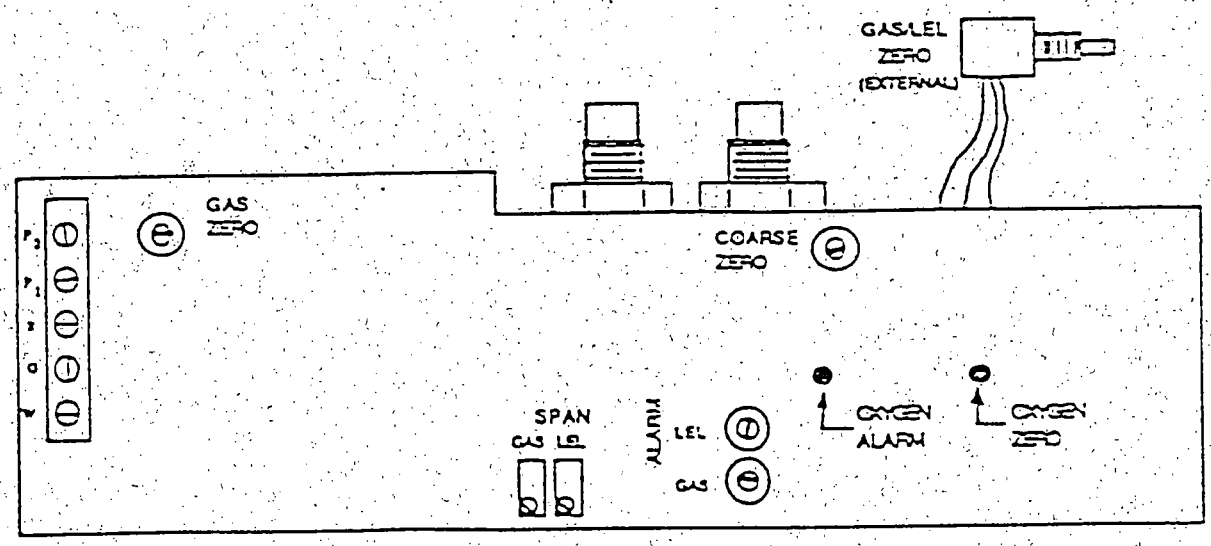

FIGURE 2

5.1.4 Turn the external GAS/LEL ZERO control to the center of its range.

5.1.5 Turn the COARSE ZERO potentiometer to bring the meter to the zero reading.

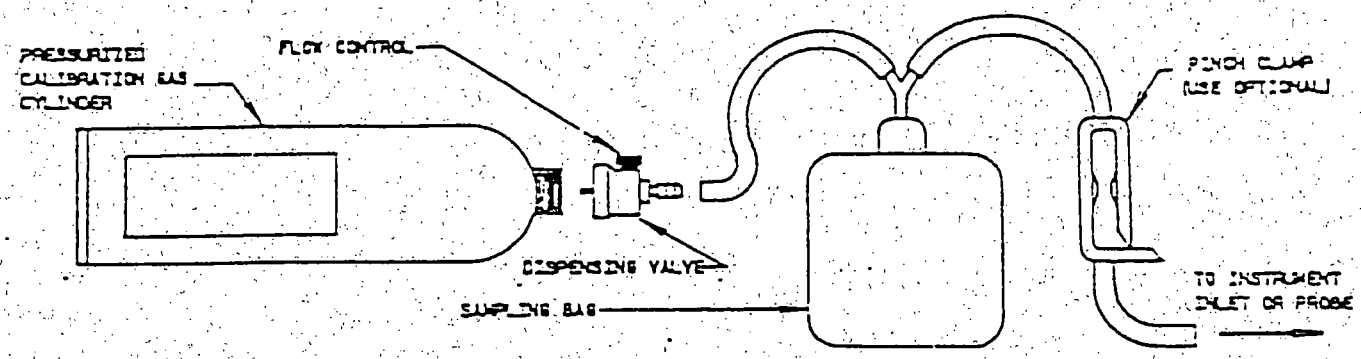

FIGURE 3

5.1.6 Remove the protective red plastic cap from the cylinder outlet of a tank of methane (2 to 5% LEL). See Figure 3.

5.1.7 Check the DISPENSING VALVE to see that the FLOW CONTROL is shut off (clockwise), but not so tightly as to damage the valve seat.

5.1.8 Screw the DISPENSING VALVE onto the cylinder outlet, being careful not to cross-thread. Tighten the valve firmly.

5.1.9 Push the short piece of flexible plastic tubing from the Y-fitting onto the barbed nipple of the DISPENSING VALVE.

5.1.10 Locate the adjustment potentiometer(s).

5.1.11 Connect the longer plastic tube with the PINCH CLAMP to the instrument. The PINCH CLAMP should be open (push latch forward to open).

5.1.12 Allow the instrument to evacuate the SAMPLING BAG until flat then close the PINCH CLAMP (squeeze paralleled faces together until tight and latched).

5.1.13 Open the FLOW CONTROL slightly so that the SAMPLING BAG begins to expand, then quickly open the PINCH CLAMP. As the instrument processes the sample, the bag will either start to expand further or to deflate. If the bag is expanding, close down on the FLOW CONTROL (clockwise), or if it is deflating, open the FLOW CONTROL slightly until the bag stays about half inflated.

5.1.14 Observe the meter on the instrument. The reading will increase, then stabilize. As soon as the reading is stable, compare with the value on the pressurized calibrating gas cylinder and record or change with the LEL SPAN potentiometer as appropriate.

NOTE Care must be taken to ensure that the LEL SPAN potentiometer and not the LEL alarm is adjusted.

5.1.15 Quickly close the FLOW CONTROL on the DISPENSING VALVE (clockwise) and disconnect the LEL calibration cylinder.

5.2 % GAS CALIBRATION

The 0 to 100% GAS range is calibrated in the same manner as the LEL range above, except adjustment is made using the GAS ZERO and GAS SPAN potentiometers and a cylinder of 100% methane (natural) gas. Place the GAS LEL switch in the 100% GAS (in) position for this calibration. **Do not change other zero adjustments.**

When the calibration is complete, remove the DISPENSING VALVE from cylinder before storage, to prevent loss of cylinder contents, then cap the cylinder. SAMPLING BAG, DISPENSING VALVE and PINCH CLAMP may be left connected to the tubing for storage.

5.3 OXYGEN CALIBRATION

5.3.1 Calibrate in fresh air.

5.3.2 Put the OXY/GAS-LEL switch in the OXY (in) position with the colored indicator showing.

5.3.3 Turn the OXY CAL potentiometer to bring the meter to the oxygen CAL mark (21%).

NOTE If problems are encountered during the calibration procedures, the unit must be replaced.

NOTE Instrument alarms are factory-set and must not be changed.

SECTION 6
MAINTENANCE/TROUBLE SHOOTING

6.1 BATTERIES

6.1.1 Check the battery voltage periodically by pressing the BATTERY CHECK switch. Charge the batteries before the voltage reaches minimum.

When connecting the charger, always follow these steps:

a. Confirm that the plug is inserted in the correct way, with the THIS SIDE UP label upwards. The plug is keyed with a non-current carrying third socket to aid in proper insertion.

b. Verify that the amber light on the charger is on. Leave connected until the green light comes on, indicating that the 16 hour charge is completed.

6.1.2 If sufficient voltage cannot be obtained after charging, the battery needs to be changed.

6.2 CATALYTIC SENSOR (LEL RANGE)

The sensor assembly may require replacement if:

a. The meter cannot be set to zero within the range of the GAS/LEL ZERO potentiometer.

b. The meter cannot be set to the desired level within the range of its SPAN potentiometer.

6.3 THERMAL CONDUCTIVITY SENSOR (GAS RANGE)

The sensor assembly may require replacement if:

a. The meter cannot be set to ZERO within the range of the GAS ZERO potentiometer.

b. The meter cannot be set to the desired level within the range of its GAS SPAN potentiometer.

6.4 OXYGEN DETECTOR

The oxygen sensor assembly may require repair if the meter cannot be set to the desired level within the range of the OXY CAL adjustment.

SECTION 7
PRECAUTIONS AND NOTES ON OPERATION

7.1 HEATED SAMPLES

When sampling spaces that are warmer than the instrument, such as hot manholes, remember that condensation can occur as the sample passes through the cooler sample line. Water vapor condensed in this way can block the flame arrestors and interfere with sensors and pump operation.

7.2 FILAMENT POISONING

Certain substances have the property of desensitizing the catalytic surface of the platinum filament. These substances are termed catalyst poisons and can result in reduced sensitivity or in failure to give a reading on samples containing combustible gas. The most commonly encountered catalyst poisons are silicone vapors. Samples containing such vapors, even in small proportions, should be avoided.

Frequent calibration checks on known-gas samples are desirable, especially if the possibility exists of exposure to silicones.

7.3 OTHER GASES AND VAPORS

The instrument is designed and calibrated specifically for detection of methane. It can be calibrated for use on other gases and vapors, by proper adjustment of the calibration control while sampling a known gas-air mixture.

7.4 RICH MIXTURES

When sampling rich mixtures, on the 100% LEL range, the following instrument action may be expected:

1. Mixtures up to 100% LEL - Reading on scale

2. Mixtures between LEL and Upper Explosive Limit (UEL) - Readings at top of meter

3. Mixtures above UEL - As sampling continues, the meter first goes to the top of the scale, then comes back down on scale. Very rich mixtures will give close to a zero reading on the catalytic sensor.

Rich mixtures should always be tested first on the 100% GAS range, to avoid any uncertainty as to actual concentration.

7.5 OXYGEN DEFICIENT MIXTURES

Samples which do not have the normal proportion of oxygen may tend to read low in the LEL range, as there is not enough oxygen to react with all the combustible gas present in the sample. As a general rule, samples containing 10% oxygen or more have enough oxygen to give a full reading on the catalytic sensor for any combustible gas sample up to the LEL. This limitation does not apply to the thermal conductivity sensor used for the 100% GAS range.

NOTE For very rich or oxygen-lean mixtures the instrument should be used in the 0 to 100% GAS range.

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EAG/dlk/MAL/H&SPC
SAFETY:IOPS

C5

SENSIDYNE GAS DETECTOR FOR CARBON DIOXIDE

BLUE BOOK

SENSIDYNE®

Gastec Precision Gas Detector System Manual

SENSIDYNE®

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I. GENERAL

1. Gas Detector Tube System

In order to insure environmental improvement it is most important to measure simply and rapidly very minute gas or vapor concentration in the work place.

Modern technology provides a variety of instruments for the measurement of hazardous gases and vapors. Such relatively sophisticated instruments must:

1. be capable of measuring concentrations in the range of threshold limit values.
2. must be easy to use, and available for taking quick samples in the work environment.
3. must produce results for evaluation and analysis within a few minutes, making it unnecessary to take samples back to the laboratory.
4. must provide dependable accuracy and reproducibility.
5. make it unnecessary to use expensive and delicate instrumentation.

The following three steps are essential for the chemical analysis of toxic gas or vapor:

1. collecting of samples
2. preparation of measuring apparatus and reagents
3. chemical treatment and analysis of the samples

The introduction of detector tube technology eliminates steps two and three above and makes it possible to achieve immediate results with the accuracy and reproducibility necessary for environmental assessment.

2. Principle of Gastec Detector Tube

Each detector tube contains a precise amount of detecting reagents in a constant inner diameter glass tube and is hermetically sealed at both ends.

To operate, break tips off a fresh tube and connect the tube to the Gastec sampling pump and pull the handle to take the required sample (sample gas). The chemical reagent in the detector tube will then react with the sample gas immediately and a color stain will develop starting at the inlet of the detector tube. The gas concentration is measured at the interface of stained-to-unstained reagent when staining stops.

Gastec detector tubes contain colorimetric reagents adsorbed on fine grain silica gel, activated alumina or other adsorbing media. The reagents are sensitive to particular gases or vapors and react quantitatively to provide a length-of-stain indication.

3. Characteristics of Gastec Gas Detector System

Detector Tube

1. Precision direct reading calibration scale printed on each tube.
2. Every tube and tube box displays the quality control number and chemical symbol. The expiration date is stamped on each box.
3. Controlled small internal diameter of tubes provide longer stain length with one pump stroke (100 ml), resulting in more accurate readings and shorter sampling times.
4. Sensitive reagents with high reaction rates provide a clear line of demarcation of color stain, for improved accuracy in tube reading.

Sampling Pump

1. Light weight, compact (approx. 10 oz.).
2. Pulls the highest vacuum (8.1" of Hg).
3. Gastec design does not require flow-rate orifices.
   Malfunction of the pump by clogging or leaking orifices is eliminated.
4. Handle locks at precise intake volume of 50 or 100 cc.
5. Friction-proof lubricant seal packing provides complete leakproof sampling at all times.

4. Accuracy of Gastec Detector Tube

Calibration Scale

The calibration curve on most Gastec detector tubes is a straight line, and points on the scale are at equal intervals (Fig. 1). Therefore, it is possible to measure the concentration below the lowest calibration scale by repeating pump strokes. It is also possible to measure the concentration above the highest calibration scale by using a half-pump stroke. This means that the measuring range of Gastec detector tubes is easily and accurately expanded by varying the number of pump strokes.

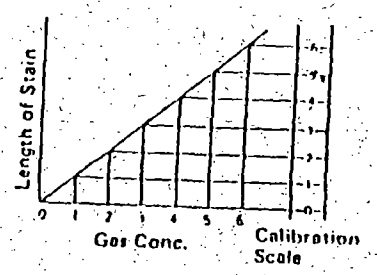


Fig. 1

Since all of the Gastec tubes are direct reading, the calibration scales are printed on the basis of individual production lots. Therefore, the variation of inner tube diameters, precision of tube packing, and the quality and reactivity of each reagent are eliminated. There is no need for extra charts or graphs whose accuracy is questionable for anything other than a single production run.

Certification of Gas Detector Tube Units

The National Institute for Occupational Safety and Health (NIOSH) has established a testing and certification program to insure that devices and instruments used in assessing and protecting against health hazards meet acceptable performance requirements. The Occupational Safety and Health Act (1970) requires the use of certified equipment unless no such equipment exists. At this point, detector tubes and sound level meters are the only instruments being tested under this program.

Following are the details of the test standard as published in the Federal Register dated May 8, 1973:

1. Every manufacturer must submit the quality control documentation including the quality control system of the company, the quality control program and the

quality control plan of the measuring apparatus to the Quality Control Section of TCL prior to the certification test. The applicant will be granted its basic qualification by the Section after the documentation check.
2. In accordance with the Federal Register, the applicant must submit the quality control documents for the detector tube to be tested, with test results certifying that the tubes satisfy the standard, and an appropriate number of tubes. After passing this check, the applicant may proceed to the next step.
3. The applicant submits the detector tubes for inspection. The inspection procedure is divided into two parts:
   1) physical inspection tests such as the longitudinal axis of glass tubes, detecting layer and calibration scale and
   2) chemical reaction tests for indicating accuracy with standard gas mixture.

   Physical inspection tests are performed during a sampling inspection in accordance with Inspection Level II as described in MIL-STD-105D. The tubes which pass the above inspection proceed to the Air Sampling Section and are tested for accuracy.
4. The accuracy test is conducted on four concentrations; one-half, one, two and five times the TLV in accordance with the Inspection Level IV as described in MIL-STD-104.

   The standard gas generation method and the standard method of analysis for the test are verified in the Microanalysis Laboratory of the National Bureau of Standards; then the concentration of the gas is confirmed by the Gas Detector Tube Standard Committee. The detector tube stained by the standard gas concentration is read by the three (3) independent tube readers. (They will read 4 end points of circumference on the longitudinal axis.) The results of the tube reading are put into the computer, and the standard deviation percentages are then calculated.
5. To be certified by NIOSH, the detector tube must satisfy the following test standards:
   1) For the accuracy test, gas detector tube units shall produce measurements of contaminant concentration with ±35% of the actual value at one-half the test standard and within ±25% of the actual value at 1, 2 and 5 times the test standard.
   2) The standard deviation of the tube readings obtained from three independent tube readers shall be within 10%.
   3) On channelling of airflow through the detector tube, the maximum variation of stain length around the circumference of the tube at the interface between stained and unstained reagents shall be within 20%.
6. Detector tubes certified under this program will be published in the Federal Register at that time, the certification number will be issued, and the government will place the item on the approved list. Thereafter, NIOSH may purchase the tubes from the market at random and test them to determine if the tubes continue to meet these exacting standards. Tubes which fail to meet the test standard will have the certification withdrawn until the defect is corrected. The following Gastec Detector Tubes have been certified by NIOSH and issued the certification number:

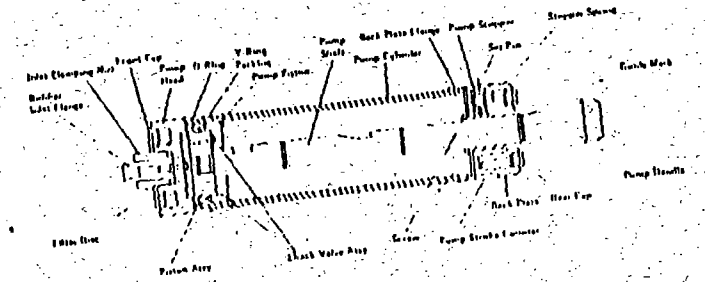
NOTE: NIOSH Certification Program has been discontinued.

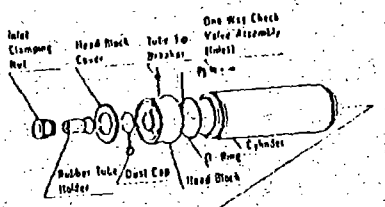
Tube No. 1La	Carbon Monixide	TC-84-014
Tube No. 5La	Sulfur Dioxide	TC-84-017
Tube No. 9L	Nitrogen Dioxide	TC-84-018
Tube No. 4LL	Hydrogen Sulfide	TC-84-020
Tube No. 2L	Carbon Dioxide	TC-84-021
Tube No. 3M	Ammonia	TC-84-023
Tube No. 134	Carbon Tetrachloride	TC-84-036
Tube No. 132H	Trichloroethylene	TC-84-038
Tube No. 8La	Chlorine	TC-84-041
Tube No. 121	Benzene	TC-84-043
Tube No. 10	Nitric Oxide	TC-84-049
Tube No. 122	Toluene	TC-84-053
Tube No. 131La	Vinyl Chloride	TC-84-060
Tube No. 12L	Hydrogen Cyanide	TC-84-068

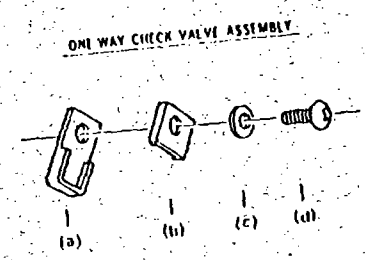
5. Gastec Pump Performance

Description of Pump

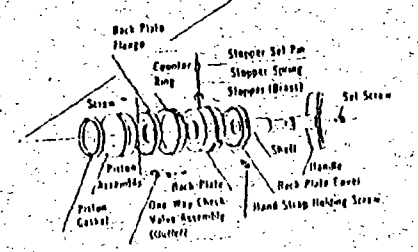
Construction of the Gastec pump is illustrated below. This pump pulls the highest vacuum for its type (8.1" of Hg). There are no flow-rate orifices to cause malfunction of the pump by clogging or leaking. A friction-proof piston gasket (lubricant seal packing) provides complete leakproof sampling at all times.







(a) Rubber valve flap
(b) Valve retainer
(c) Plain washer
(d) Valve screw



Checking Pump Performance

A. Visually check rubber inlet flange for cracks or tears. Replace if damaged. Tighten inlet clamping out.

B. Valve Leak Check

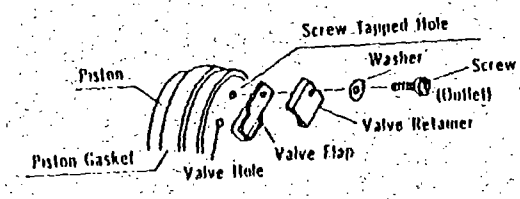
1. Insert a fresh sealed detector tube into pump. Misalign red dots on pump and handle. Pull several fairly rapid continuous full pump strokes.
2. Pull handle out 6 mm (1/4 inch) and hold in this position for 1 or 2 seconds.
3. Release handle.
4. If handle return to within 1.5 mm (1/16 inch) or less of fully closed position, continue to step C.
5. If handle does not return to within 1.5 mm (1/16 inch) of fully closed position (or less), perform the following Valve Lubrication instruction outlined below.

C. Field Volume Check

1. Insert a fresh sealed detector tube into pump.
2. Align Guide Marks on pump body and handle.
3. Pull handle firmly and at a moderate speed until handle locks into position. Wait 1 minute.
4. Unlock handle by turning it and guide it back.
   TO PROTECT PUMP STOPPER from breakage, do not release the handle and allow it to spring back when conducting a leak test. Make sure you hold your hand onto the handle and guide it back.
5. Pump handle should return to within 6 mm (1/4 inch) or less of the fully closed position.
6. If pump handle does not close to within 6 mm (1/4 inch) or less, follow lubrication instructions and retest.

D. Lubrication Instructions (Perform Laboratory Volume Check "E" after each lubrication)

1. Valve Lubrication
   a. Unscrew back plate and withdraw piston from pump cylinder.
   b. Remove check valve from piston.
   c. Clean valve and piston with lint-free cloth. Proper valve cleaning is as follows:
      Place cloth flat on desk.
      Wipe rubber valve flap in a flat position across cloth.
      Do not bend the rubber flap valve.
   d. Apply a small amount of grease evenly around the valve opening to form a thin film. A thin film is nearly invisible.
   e. Replace valve assembly loosely in the same manner as removed.



f. Before tightening the screw, align valve so that valve hole is in center of valve flap.

g. Then push the rectangular valve retainer all the way toward loose end of valve falp.

h. Now tighten screw. If a torque driver is available, tighten to 0.8 Kg-cm. Otherwise, be careful not to overtighten screw. When tightened, screw must not deform rectangular valve retainer.

2. Piston Gasket Lubrication

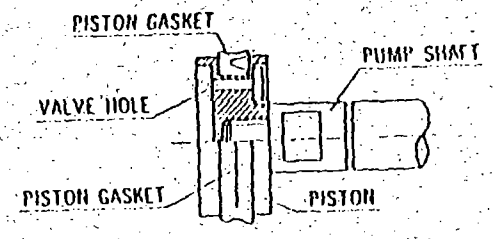
a. Wipe off piston and cylinder with a clean lint-free cloth.

b. Remove piston gasket with a small bladed screwdriver. Take care not to cut gasket.

c. Clean slot in piston with lint-free cloth. Wipe off rubber gasket.

d. Wipe an ample supply of grease into gasket slot on piston and inside gasket.

e. Replace gasket making sure that open side of gasket is toward pump handle.



f. With the excess grease from piston slot, wipe around outside of gasket and piston.

g. Wipe an ample amount of grease into cylinder at the area of piston entrance.

h. Insert piston slowly into the cylinder. Work the piston back and forth slowly in the cylinder several times.

i. Now screw back plate firmly onto cylinder.

j. Repeat leak tests.

k. If any leak remains, replace piston gasket.

l. Only if a leak persists, go to procedure below.

3. Pump Head Lubrication

a. This is only necessary where all previous procedures have failed to correct a leak.

b. Visually check pump head "O" ring for cracks.

c. Replace "O" ring if cracked.

d. Place a light coat of grease on pump cylinder head screw threads and the "O" ring.

e. Insert new "O" ring.

f. Screw pump head firmly on to "O" ring and make sure "O" ring is seated uniformly. Overtightening pump head may push "O" ring out of place. Do not overtighten.

g. Wipe off excess grease.

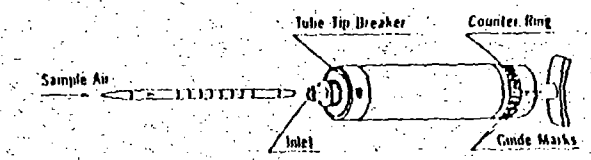
E. Laboratory Volume Check (To be performed at least once after each lubrication)

The Gastec pump can be checked periodically to assure that 100 ± 5 ml are being sampled.

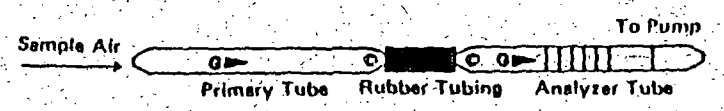
1. Arrange a graduated 100 ml soap film flow meter in a volume test mode.
2. Insert a fresh Gastec tube into the pump. The tube must be broken at both ends (ready for use).
3. Attach the Gastec tube to top of soap film flow meter with rubber hose. Make sure there are no leaks.
4. Pull pump handle out full to lock at one stroke in normal sampling manner.
5. Wait until the bubble stops moving and read the volume evacuated.
6. If the volume evacuated is other than 100 ± 5 ml, proceed to lubrication instruction and retest.

6. Operating Procedure for Gastec Precision Gas Detector System

Sampling & Measurement Procedure



1. Break tips off a fresh detector tube by bending each tube end in the tube tip breaker of the pump.
2. Insert tube securely into pump inlet with arrow on tube pointing toward pump.
3. For twin tubes, connect Ⓒ marked ends with rubber tubing after breaking each end. Insert analyzer tube into pump with arrows on tubes pointing toward pump. See figure below.



4. Make certain pump handle is all the way in. Align guide marks on pump body and handle.
5. Pull handle out to desired stroke volume. Handle can be locked on either 1/2 pump stroke (50 ml) or 1 pump stroke (100 ml).
6. Read concentration at the interface of stained-to-unstained reagent when staining stops. Unlock handle by making 1/4 turn and return it to starting position.
7. In case more pump strokes are indicated in the instruction sheet included in each box of tubes, take additional sample by repeating pump strokes without removing tube.

Correction for Temperature, Humidity or Pressure:

Calibration of the Gastec detector tubes is based on a tube temperature of 20°C

(68° F), approximately 50% relative humidity, and normal atmospheric pressure.

1. No correction is normally required for tube temperature of 0° – 40° C (32° – 104° F) and for relative humidity range of 20 – 90%.
2. Where the detecting reagent is abnormally sensitive to temperature or humidity, a correction table or chart is provided in each box of tubes. In this case, the tube reading must be corrected using correction table or chart.
3. Tube reading is proportional to absolute pressure. To correct for pressure, multiply by

$$\frac{760}{\text{Atmospheric Pressure (mm Hg)}}$$

How to Use Counter Ring:
In case numerous pump strokes are required, use the counter ring of the pump.
(1) Turn the counter ring manually counterclockwise after each pump stroke.
(2) The operator may thus keep count of the number of pump strokes.

For more details see the instruction sheet included in each box of detector tubes.
Detector tube should be stored in accordance with instructions on each box.

7. Different Methods of Sampling

1. Effect of sampling time and related data

Concentration of contaminants and chemical substances in the air is always fluctuating. On the other hand, sampling is usually conducted within certain time and frequency limits. Therefore, a deviation naturally exists between the average environmental concentration and results obtained on the basis of "grab" samples.

For example, Fig. 2 below shows time-lapse variation of dust concentration which was observed by using Tyndallometer readings plotted in 30-second intervals. This test was conducted at a raw material mixing plant in the glass mill.

The variation of concentration during the hour was up to 60 times the average concentration of dust. This idea can be adapted to the gas concentration in air of toxic substances as follows:

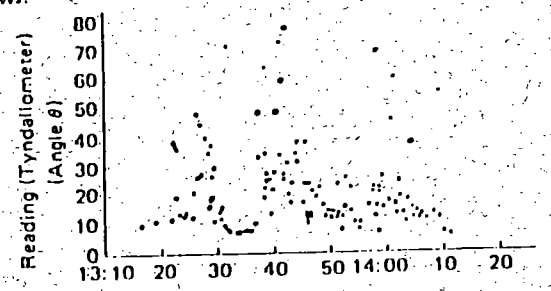


Fig. 2 Time-lapse Variation of Dust Concentration
(Place: Raw material mixing plant in glass mill)
by Tyndallometer

The variation of the measured concentration is largely dependent on the sampling time and position. Large differences occur when the operation is intermittent. If the sampling time is short and the quantity of sample gas or vapor is relatively small, large variations will result between instantaneous concentration and average concentration. Under extremely adverse conditions, there can be as much as ten fold difference in detector tube readings taken at different times in the same location.

2. Tanks, Manholes, or Closed Places

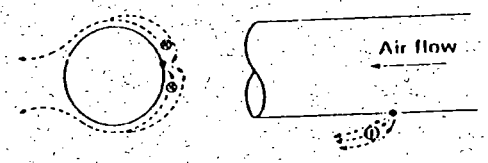
It is dangerous to enter a manhole directly without a pre-check. Before entering any such closed place, the workman must first check the gas level by using an extension hose in at least three levels, that is the higher, middle, and lower level of the closed area. The reason for this is as follows:

$$\text{Specific Gravity of Gas} = \frac{\text{Molecular Weight of Gas}}{\text{29 (Average MW of Air)}}$$

If the total fraction is greater than 1.0, it means the gas will rise to the top. However, if the total fraction equals more than 1.0, it means the gas concentration is increasing at the lower level.

3. Leak-check from Pipeline

If an air flow or a breeze is observed prior to taking the measurement, measuring points must be moved away from the direction of the air flow.



- - - - - - : Gas flow
⊗ : Measuring points

Fig. 3

Caution: If the measurement is conducted at a point some distance from the pipeline, the gas emitted will be diluted very rapidly, and the true concentration of the gas cannot accurately be detected. Therefore, measurement must be conducted at a point as close as possible to the suspected leak.

4. Measurement in a Chemical Plant, Gas Concentration in Pipelines

It is sometimes necessary to collect the gas sample temporarily in a sampling bag or a bottle as shown in the two examples below. Measurement of the concentration is made by passing a sample through an appropriate Gastec tube.

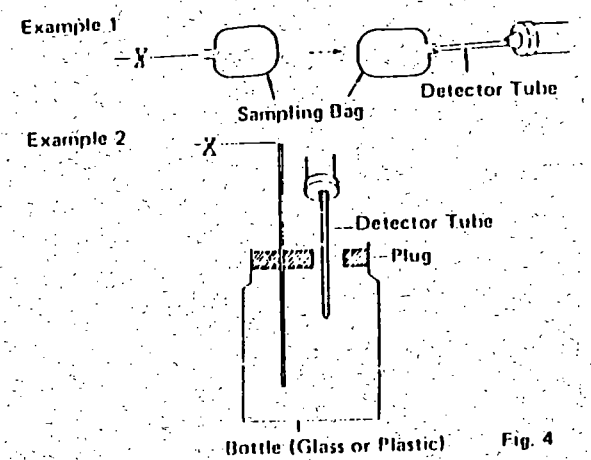


Fig. 4

5. Measurement and Threshold Limit Value of Liquid Mixture

A rapid reliable method for measuring concentration of industrial solvents in air is available using the Gastec Precision Gas Detector System.

5-1 Elements Evaporating from Solvent

Industrial solvents are liquids but have an appreciable vapor pressure at room temperature. The composition of the vapor arising from the solvent can be quite different from the composition of the solvent itself.

Take, as an example, a lacquer thinner mixture of 66% of toluene, 14% each of ethyl acetate and butyl acetate, 4% of butyl alcohol and 2% of cellsolve. At the early stage of its evaporation, the lower-boiling point metarial vaporize.

If a coating plant uses the above thinner, the thinner will vaporize according to the chart below.

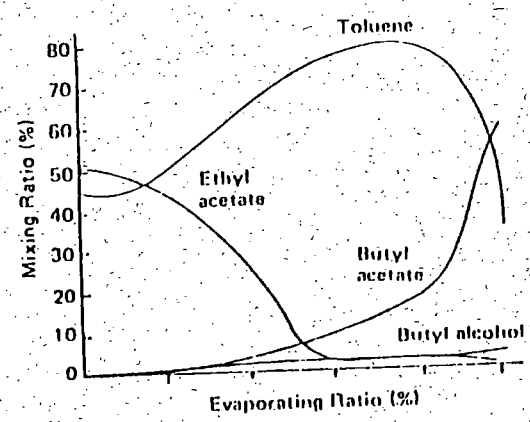


Fig. 5 Evaporation from Mixing Solvent

The ratio of vaporized components is greatly dependent upon the working environment even if the same solvents are used. Therefore we must carefully consider the environmental assessment of exposure to complex mixtures. Many lacquer thinner manufacturers keep the ratio of materials secret. However, OSHA has legislated that each producer must publish the principal components and hazardous properties of all proprietary solvents in Material Safety Data Sheets.

5-2 Selecting Measuring Apparatus

One of the best methods used in measuring each component of a solvent mixture is by means of gas chromatography. However, a simpler analysis using detector tubes is usually preferred for daily environmental measurement. Use of detector tubes and selection of tubes is greatly dependent on the individual case. It is recommended that a specialist be consulted for correct interpretation of the results. Items such as interferences and reaction rates have a major effect on the accuracy and precision of the measurement.

5-3 Threshold Limit Value for Mixtures

When two or more hazardous substances are present, their combined effect, rather than that of either individually, would be given primary consideration. In the absence of information to the contrary, the effects of the different hazards should be considered as additive. That is, if the sum of the following fractions,

$$\frac{C_1}{T_1} + \frac{C_2}{T_2} + \cdots\cdots \frac{C_n}{T_n} = 1$$

exceeds one, then the threshold limit of the mixture should be considered as being exceeded. C_1 indicates the observed atmospheric concentration, and T_1 the corresponding threshold limit (see example below).

It is essential that the atmosphere be analyzed both qualitatively and quantitatively for each component present, in order to evaluate compliance or noncompliance with this calculated TLV.

$$\frac{C_1}{T_1} + \frac{C_2}{T_2} + \cdots\cdots \frac{C_3}{T_3} + \cdots\cdots = 1$$

Example: Air contains 5 ppm of carbontetrachloride (TLV = 10 ppm), 20 ppm of 1, 2-dichloroethane (TLV = 50 ppm) and 10 ppm of 1, 2-dibromoethane (TLV = 20 ppm)

Atmospheric concentration of mixture = 5 + 20 + 10 = 35 ppm of mixture

$$\frac{5}{10} + \frac{20}{50} + \frac{10}{20} = \frac{25+20+25}{50} = 1.4$$

Threshold limit is exceeded. Furthermore, the TLV of this mixture may be calculated by reducing the fraction to 1.0; i.e.

$$\text{TLV of mixture} = \frac{35}{1.4} = 25 \text{ ppm}$$

When a given operation or process characteristically emits a number of harmful dusts, fumes, vapors, or gases, it will frequently be only feasible to attempt to evaluate the hazard by measurement of a single substance. In such cases, the threshold limit used for this substance should be reduced by a suitable factor, the magnitude of which will depend on the number, toxicity and relative quantity of the other contaminants ordinarily present.

Examples of processes which are typically associated with two or more harmful atmospheric contaminants are welding, automobile repair, blasting, painting, lacquering, certain foundry operations, diesel exhausts, etc.

II Gastec Detector Tubes

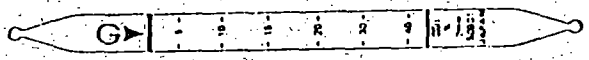
These definitions apply to each of the following pages.

1. Performance of the Tube (n: Number of Pump Strokes)
2. Detecting Principle of the Gas
3. Interferences of the Tube
4. Method of Standard Gas and Analysis
   - 4.1 Standard Gas and Analysis
   - 4.2 Method of Analysis
5. Dangerous and Hazardous Properties
   - 5.1 T.L.V.-T.W.A.: The timeweighted average concentration for a normal 8-hour workday or 40-hour workweek, to which nearly all workers may be repeatedly exposed, day after day, without adverse effect.
     Threshold Limit Value-Time Weighted Average for 1985 adopted by American Conference of Governmental Industrial Hygienists.
   - 5-2 T.L.V.-STEL: The maximum concentration to which workers can be exposed for a period up to 15 minutes continuously without suffering from 1) intolerable irritation, 2) chronic or irreversible tissue change, or 3) narcosis of sufficient degree to increase accident proneness, impair self-rescue, or materially reduce work efficiency, provided that no more than four excursions per day are permitted, with at least 60 minutes between exposure periods, and provided that the daily TLV-TWA also is not exceeded.
     Threshold Limit Value-Short Term Exposure Limit for 1985 adopted by American Conference of Governmental Industrial Hygienists.
   - 5-3 F.L.: Flammable Limits in Air (lower and upper limits)
6. Physical Constants
   - 6-1 C.F.: Chemical Formula
   - 6-2 M.W.: Molecular Weight
   - 6-3 S.G.: Specific Gravity, Gas @20°C (68°F), NTP 1 atm unless otherwise stated
   - 6-4 M.P.: Melting Point
   - 6-5 B.P.: Boiling Point
   - 6-6 AIT: Autoignition Temperature
   - 6-7 V.P.: Vapor Pressure (mmHg)
7. Application for other gases – Other gases for with the same detector tube may be used to detect.

CARBON DIOXIDE EXTRA HIGH RANGE TUBE

No. 2HH

1. Performance



Calibration Scale	5 - 40%	(n=1/2)	Color Change	White - Purple
Measuring Range	2.5 - 5% 5 - 40%	(n=1) (n=1/2)	Sampling Time	1 min./pump stroke
Detecting Limit	0.5%	(n=1)	Shelf Life	3 years

2. Detection Principle

Carbon dioxide reacts with hydrazine to form carbonic acid monohydrazid, which discolors redox indicator (crystal violet).

$CO_2 + N_2H_4 \rightarrow NH_2 \cdot NH \cdot COOH$

3. INTERFERENCES:

Other gases and vapors do not affect the tube reading

4. 4.1 Standard Gas Generation Method: Cylinder bottle gas & dynamic gas flow system

4.2 Method of Analysis: Gas chromatography (TCD)

5. Dangerous & Hazardous Properties

	Concentration	Phisical Effects
T.L.V.: 5000 ppm	2.5%	No effect for a few hours
STEL: 15000 ppm	10%	Can endure for a few minutes
F.L.:	12 – 15%	May cause death in exposures for several hours

6. Physical Constants

C.F.: CO_2 M.W.: 44.01 S.G.: 1.53
M.P.: −56.6°C B.P.:
AIT: V.P.:

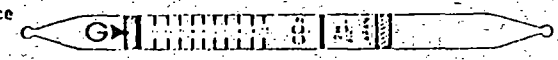
7. Application for other gases

8510

CARBON DIOXIDE HIGH RANGE TUBE

No. 2H

1. Performance



Calibration Scale	1.0 - 10%	(n=1)	Color Change	White - Purple
Measuring Range	0.5 - 1.0% 1.0 - 10% 10 - 20%	(n=2) (n=1) (n=1/2)	Sampling Time	1 min./pump stroke
Detecting Limit	0.1%	(n=2)	Shelf Life	3 years

2. Detection Principle

Carbon dioxide reacts with hydrazine to form carbonic acid monohydrazide, which discolors redox indicator (crystal violet).

$CO_2 + N_2H_4 \rightarrow NH_2 \cdot NH \cdot COOH$

3. Interferences:
Other gases and Vapors do not affect the tube reading.

4. 4.1 Standard Gas Generation Method: Cylinder bottle gas & dynamic gas flow system

4.2 Method of Analysis: Gas chromatography (TCD)

5. Dangerous & Hazardous Properties

	Concentration	Physical Effects
T.L.V. = 5,000 ppm	2.5%	No effect for a few hours
STEL = 15,000 ppm	10%	Can endure for a few minutes
F.L. =	12 – 15%	May cause death in exposures for several hours

6. Physical Constants

C.F.: CO_2 M.W.: 44.01 S.G.: 1.53 M.P.: −56.6°C B.P.: −78.5°C
AIT: V.P.:

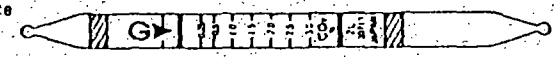
7. Application for other gases

8301

CARBON DIOXIDE LOW RANGE TUBE

No. 2L

1. Performance



Calibration Scale	0.25 – 3.0% (n=1)	Color Change	White - Purple
Measuring Range	0.13 – 0.25% (n=2) 0.25 – 3.0% (n=1) 3.0 – 6.0% (n=½)	Sampling Time	2 minutes/pump stroke
Detecting Limit	0.025% (n=2)	Shelf Life	3 years

2. Detection Principle

Carbon dioxide reacts with hydrazine to form carbonic acid monohydrazide, which discolors redox indicator (crystal violet).

$$CO_2 + N_2H_4 \longrightarrow NH_2 \cdot NH \cdot COOH$$

3.

Interferences:
Other gases and vapors do not affect the tube reading.

4. 4.1 Standard Gas Generation Method: Cylinder bottle gas & dynamic gas flow system

4.2 Method of Analysis: Gas chromatography (TCD)

5. Dangerous & Hazardous Properties

T.L.V. = 5000 ppm
STEL = 15000 ppm
F.L. =

Concentration	Physical Effects
2.5%	No effect for a few hours
10%	Can endure for a few minutes
12 – 15%	May cause death in exposures for several hours

6. Physical Constants

C.F.: CO_2 M.W.: 44.01 S.G.: 1.53 M.P.: -56.6°C B.P.:
AIT: V.P.:

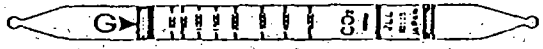
7. Application for other gases

8201

CARBON DIOXIDE EXTRA LOW RANGE TUBE

No. 2LL

1. Performance



Calibration Scale	300-5000 ppm (n=1)	Color Change	White - Purple
Measuring Range	100 - 300 ppm (n=3) 300-5000 ppm (n=1) 4600-11500 ppm (n=1/2)	Sampling Time	2 min./pump stroke
Detecting Limit	30 ppm (n=1)	Shelf Life	3 years

2. Detection Principle

Carbon dioxide reacts with hydrazine to form carbonic acid monohydrazide, which discolors redox indicator (crystal violet).

$$CO_2 + N_2H_4 \rightarrow NH_2 \cdot NH \cdot COOH$$

3.

Interferences	Concentration	Result	Comment
Ammonia	Up to 1,000 ppm	No effect	At more than 1,000 ppm gives minus error
Carbon monoxide	Up to 500 ppm	"	
Sulfur dioxide	Up to 30 ppm	"	
Nitrogen dioxide	Up to 30 ppm	"	
Chlorine	Up to 20 ppm	"	

4. 4.1 Standard Gas Generation Methods: Cylinder bottle gas & dynamic gas flow system

4.2 Method of Analysis: Gas chromatography (TCD)

5. Dangerous & Hazardous Properties

T.L.V. = 5000 ppm
STEL = 15000 ppm
F.L. =

Concentration	Physical Effects
2.5%	No effect for a few hours
10%	Can endure for a few minutes
12 – 15%	May cause death in exposures for several hours

6. Physical Constants

C.F.: CO_2 M.W.: 44.01 S.G.: 1.53 M.P.: -56.6°C B.P.:
AIT: V.P.:

7. Application for other gases

7803

C6

HNU Photoionization Detector for VOC Screen

INSTRUMENT OPERATION PROCEDURE

HNu Model PI101 Photoionization Detector

For the Detection of Organic Vapors
Through Photoionization

January, 1991

INSTRUMENT OPERATING PROCEDURE
HNU TRACE GAS ANALYZER MODEL PI 101

SCOPE AND APPLICATION

The Trace Gas Analyzer (See Figure 1), is a portable instrument used to detect, measure, and provide a direct reading of the concentration of a variety of toxic gases in the air. The analyzer employs the principle of photoionization. The sensor consists of a sealed ultraviolet light (UV) source that emits photons with an energy level high enough to ionize many trace species, particularly organics.

The analyzer consists of a probe, a readout assembly, and a battery charger. The analyzer has a concentration range from 0 PPM to 2000 PPM.

Reference:
Instruction Manual Portable Photoionization Analyzer Model PI 101.

Apparatus:
HNu Model PI 101

Battery Charger

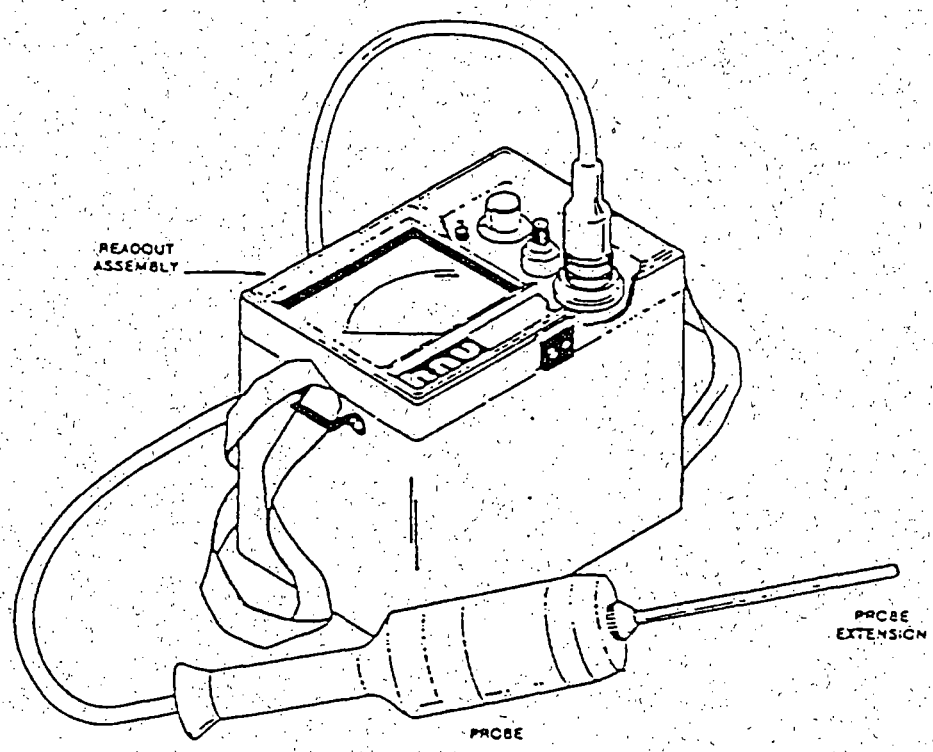
Probe options: 9.5 eV, 10.2 eV or a 11.7 eV UV lamps.

Calibration Kit - Kit containing a gas cylinder of isobutylene at concentrations of 52.1 PPM and 59.7 PPM (check the concentration marked in the cylinder provided) for the 10.2 and 11.7 eV probe respectively, and a gas regulator.

1.0 DETAILED DESCRIPTION

1.1 Meter

Indications of the instrument are displayed on a meter, visible through a window on the top face of the instrument case (See Figure 2). A mark on the scale, "BATT CK", represents the minimum permissible battery voltage, as an indication of the state-of-charge of the battery. The meter face has a scale from 0 to 20.



TRACE GAS ANALYZER

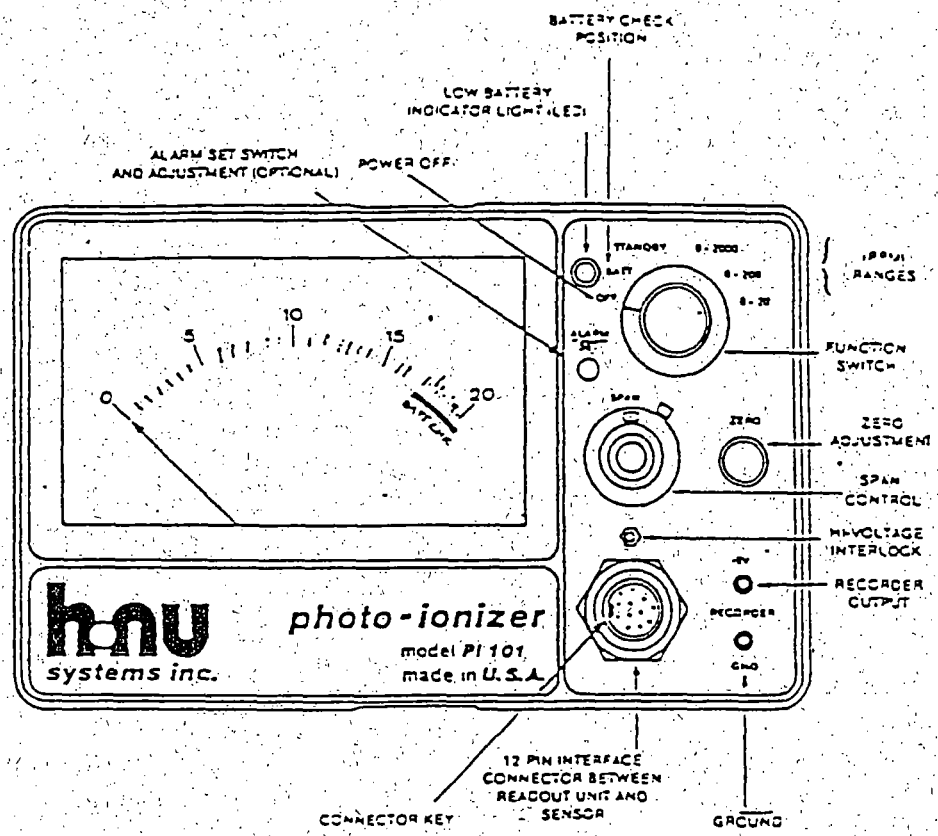
FIGURE 1

1.2 Controls and Indicators

The control and indicators are located on the front panel of the readout assembly (See Figure 2) and are as follows:

1.2.1 FUNCTION switch, a switch with six functions: OFF, BATT, STANDBY, and ranges of 0-2000, 0-200, and 0-20.

In the OFF position all operations are OFF; in the BATT position, checks the condition of the battery; in the STANDBY, all electronics are ON but the UV light source is OFF.



CONTROLS AND INDICATORS

FIGURE 2

1.2.2 ZERO adjustment, with the Function Switch in the STANDBY position, this potentiometer is used to adjust the reading to zero.

1.2.3 SPAN, this vernier potentiometer is used to set the gain of the amplifier to give direct readings of the trace gas concentrations in ppm. The whole number of the setting appears in the window of the control, decimal appears on the dial. A lock secures it at a specific setting.

1.2.4 HI-VOLTAGE, this is a normally open microswitch. Switch is open when cable not connected. Switch is automatically closed when the cable is attached. This switch may also be closed manually during maintenance checks of the readout assembly without the probe cable attached.

1.2.5 ALARM SET (optional), a potentiometer that turns the audible alarm ON and OFF and sets the ppm level at which the alarm sounds.

1.2.6 LOW BATTERY INDICATOR LIGHT, illuminates when the battery is discharged, indicates need for recharge. Readings may be taken while the unit is being recharged.

1.2.7 RECORDER (optional), provides a record of readings while analyzer operates unattended.

2.0 OPERATING PROCEDURES

The following procedures are to be used in operating the analyzer:

2.1 Unclamp the cover from the main readout assembly.

2.2 Attach the handle to the front part of the probe.

2.3 Connect the probe cable plug to the 12 pin keyed socket on the readout assembly panel (Figure 1). Carefully match the alignment slot in the plug to the key in the connector. Screw down the probe connector until a distinct snap and lock is felt.

2.4 Screw the probe extension into the probe end cap. The probe may be used without the extension if desired.

2.5 The SPAN control is set when the instrument is calibrated, refer to Section 3 below. Do not move this knob unless you are calibrating the instrument.

2.6 Turn the function switch to the BATT (battery check) position. The needle on the meter will go to the green zone if the battery is fully charged. If the needle is below the green arc or the Low Battery Indicator comes on, the battery must be recharged before the analyzer is used.

2.7 Turn the function switch to the STANDBY position. Turn the zero adjustment until the meter needle is at zero.

2.8 Turn the function switch to the appropriate operating position. Start with the 0-2000 ppm position and then switch to the more sensitive ranges. The UV light should be ON, confirmed by briefly looking into the probe to observe the purple glow from the lamp.

WARNING

Do not look at the light source closer than 6 inches with unprotected eyes.

Check that the fan is working properly by using a "Sharpie" marking pen; put the probe extension close to the tip of the pen and observe the needle deflect to the right. If the fan is not working return analyzer for repair.

2.9 The analyzer is now operational.

2.10 Hold the probe so that the extension is at the point where the measurement is to be made

---

WARNING

The instrument measures gases in the vicinity of the operator working and breathing zone and a high reading when measuring toxic or explosive gases should be cause for action for operator safety.

2.11 Take the readings as desired taking into account that air currents, or drafts or power lines in the vicinity of the probe tip may cause fluctuations in readings. Change the ranges as required.

2.12 After completion of use, check battery condition as described in paragraph 2.6.

2.13 Turn function switch to OFF position.

2.14 When not operating, leave analyzer in assembled condition, and connected to battery charger.

2.15 When transporting, disassemble probe and extension from readout assembly and return equipment to its stored condition.

2.16 Calibrate daily as indicated in Section 3.

3.0 CALIBRATION

The PI 101 Analyzer is calibrated at HNU Systems with certified standards of benzene, vinyl chloride and isobutylene. The analyzer is calibrated in the field with a certified standard of isobutylene. The analyzer is designed for operation at ambient conditions and therefore the gas standards used for calibration should be delivered to the analyzer at ambient temperatures and pressure and at the proper flow rate.

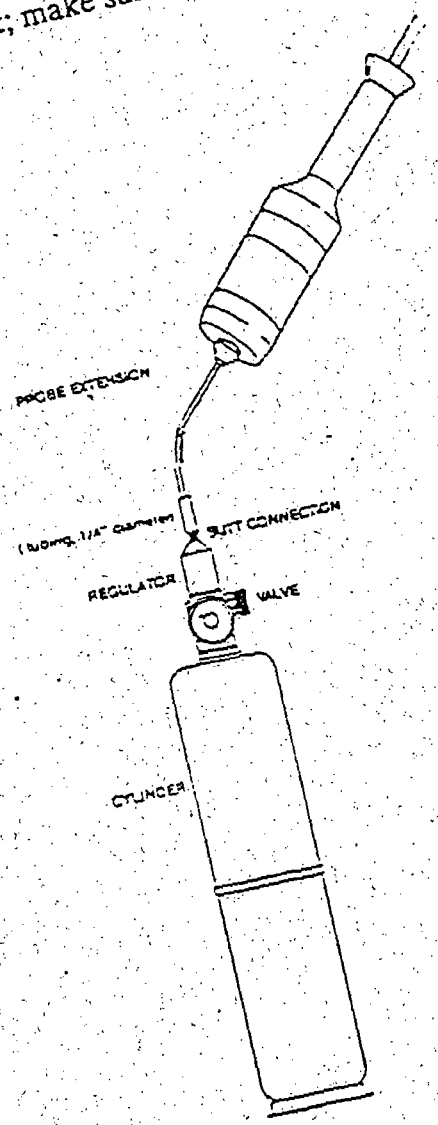
3.1 Calibration Procedure

3.1.1 Turn the function switch to BAT. The needle should be in the green region. If not, recharge the battery.

3.1.2 Turn the function switch to STANDBY. In this position the the lamp is OFF and no signal is generated. Set the zero point with the ZERO set control.

3.1.3 Turn the function switch to the 0 -20 or 0-200 range. Adjust the SPAN control setting to 9.8 when using the 10.2 eV lamp and to 5.0 when using the 11.7 eV lamp. For calibration on this range only one gas standard is required.

3.1.4 Connect the calibration gas as shown in Figure 3. The small cylinder included with the HNU unit contains Isobutylene gas standard. With the 10.2 eV lamp, the concentration should be 52.1 PPM and with the 11.7 eV lamp the concentration should be 59.7 PPM. NOTE: Normally the cylinders indicate what concentrations to expect; make sure to check this before calibration.



CALIBRATION TEST SET UP

FIGURE 3

Turn the function switch to the range position and note the meter reading. Adjust the SPAN control setting as required to read the ppm concentration of the standard. Recheck the zero setting (step 3.1.2). If readjustment is needed, repeat step 3.1.4.

3.1.5 For calibration on the 0-2000 range, use of two standards is recommended. First calibrate with the higher standard using the SPAN control for setting. Then calibrate with the lower standard using the ZERO adjustment. Repeat these several times to ensure that a good calibration is obtained.

3.1.6 If the span setting resulting from calibration is 0.0 or if calibration cannot be achieved, the lamp then must be cleaned (see section 4).

3.1.7 Shut off the cylinder as soon as the reading is established.

3.1.8 The analyzer is ready to take measurements.

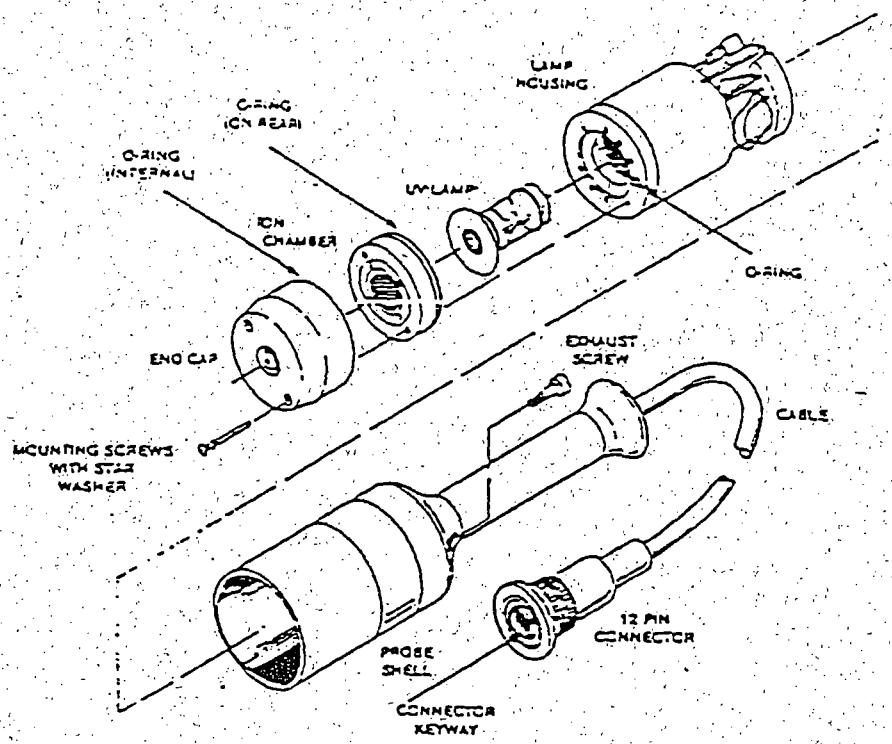
4.0 MAINTENANCE

Maintenance of the analyzer consists of cleaning the lamp, the ion chamber and the fan. During operation of the analyzer, dust, moist or other foreign matter can be drawn into the probe forming deposits on the surface of the UV lamp or in the ion chamber. This condition is indicated by meter readings that are low, erratic, unstable, non-repeatable, or drifting, or show apparent moisture sensitivity.

4.1. UV Lamp and Ion Chamber Cleaning.

Disassemble the probe by first removing the exhaust screw at the base of the probe adjacent to the handle (See Figure 4). Grasp the end cap in one hand and the probe shell in the other, gently pull to separate the end cap and the lamp housing from the shell.

Hold the lamp housing with the black end cap upright. Loosen the screws on the top of the end cap, separate the end cap and ion chamber from the lamp and lamp housing.



PROBE ASSEMBLY

FIGURE 4

CAUTION

Care must be taken so that the ion chamber does not fall out of the end cap or the light source does not fall out of the lamp housing.

Turn the end cap over in the hand. Tap lightly on the top. The ion chamber should fall out the end cap into the hand. Place one hand over the top of the lamp housing and tilt slightly. The light source will slide out of the housing.

Exercise great care in doing so to prevent inadvertent damage to these components.

4.1.1 For the 9.5 and 10.2 eV lamps:

a. First clean by rubbing gently with lens tissue dipped in a detergent solution. If this does not remove deposit, apply a small amount of HNU cleaning compound (PA101534) directly onto the lens of the lamp and spread evenly over surface with a non-abrasive tissue.

b. Wipe off compound with a new tissue.

c. Rinse with warm water or damp tissue to remove all traces of grit or oils and any static charge that may have built up on the lens. Dry with a new tissue.

d. Reinstall lamp in the detector and check analyzer operation. If performance is still not satisfactory replace the lamp.

4.1.2 For the 11.7 eV lamp:

a. Clean by putting a freon or chlorinated organic solvent on a tissue and rubbing gently.

b. **DO NOT CLEAN THIS LAMP WITH WATER OR ANY WATER MISCIBLE SOLVENTS (such as methanol or acetone). IT WILL DAMAGE THE LAMP.**

c. **DO NOT USE THE CLEANING COMPOUND USED FOR THE 9.5 OR 10.2 eV LAMPS.**

4.2 Ion chamber Cleaning.

Inspect the ion chamber for dust or particulate deposits. If such a matter is present, the chamber can be cleaned by removing the outer Teflon ring, and the four screws holding the retaining ring. Carefully remove the retaining ring aside (Note: this ring is soldered) and remove the screen. A tissue or cotton swab, dry or wetted with methanol, can be used to clean any deposits. Let the liquid dry before reassembly.
Reassemble the probe and check analyzer operation. If performance is still not satisfactory replace the lamp.

4.3 Fan Cleaning

Remove the pogo contacts and the retaining screw shown in Figure 5.

a. Lift up top supporting fan and slowly pull fan out. CAUTION: the fan does not come out all the way; it is attached to the wires.

b. Blow into the fan to remove any dust particles. If the fan does not move freely, check for any larger particles that can be removed without damaging the impellor rotor or the blades.

c. Check for wiring connections at fan motor and at probe cable connector (J3 pins A and C).

Reassemble the probe by first sliding the lamp back into the lamp housing. Place the ion chamber on top of the lamp housing, making sure that the contacts are properly aligned. The ion chamber fits only one way.

Reassemble the probe by first sliding the lamp back into the lamp housing. Place the ion chamber on top of the lamp housing, making sure that the contacts are properly aligned. The ion chamber fits only one way.

Place the end cap on top of the ion chamber and replace the two screws. Tighten the screws only enough to seal the O-ring.

CAUTION

Do not over-tighten these screws.

Line up the pins (pogo contacts) on the base of the lamp housing with the pins inside the probe shell. Gently slide the housing assembly into the probe shell. The end cap should meet the probe shell evenly after final assembly. If not, the ion chamber may be installed wrong.

D

D

LABORATORY CUSTODY SOPs

D1 Warzyn
D2 Warzyn Laboratory Waste Lab Packaging
D3 Industrial Environmental Analytical (IEA)

D1

WARZYN

CHAIN-OF-CUSTODY, LOG-IN AND TRACKING PROCEDURES

Scope and Application: This procedure is applicable to the log-in of samples and addresses such areas as: sample custody, log-in, labeling and preservation of samples. This procedure begins as the samples enter the laboratory and follows the samples to their destruction.

Procedure:

1. Chain-of-Custody Procedure: Samples arriving in the laboratory are accompanied by some type of custody record. The most common type of record is that shown in Figure 1. Follow the instructions below for all levels of chain-of-custody.

   A. Superfund Level Chain-of-Custody:

      1. Refer to the "Chain-of-Custody - Superfund Level" SOP.

   B. Routine Chain-of-Custody:

      1. Samples arriving in the laboratory are accompanied by a chain-of-custody record (refer to Figure 1).

      2. Review the custody form with the samples received and see that the spaces marked #1 - #12 have been accurately completed by the sampler.

      3. Any discrepancies with the chain-of-custody record or the samples should be immediately brought to the attention of the customer service relations person so that the problems can be solved in an efficient manner.

      4. If samples were shipped, note the courier name in the remarks section (#15) of the chain-of-custody record.

      5. Record any other comments such as: temperature upon receipt, preservation (if done in lab), problems, etc, in the remarks section (#15).

      6. Fill in the lab numbers in the section marked #16 (refer to the log-in portion of this procedure for how to obtain lab numbers).

      7. Sign and date the chain-of-custody record (#13 - #14).

      8. Route the custody record to the data management office when log-in is complete.

NOTE: The laboratory is a secured area with strict limited access. Any samples arriving to the laboratory during "off hours" are stored in the laboratory refrigerator until they are logged in. Laboratory personnel are responsible for the care and custody of the samples during the analysis stage and until the samples are removed from the refrigerator and properly disposed of.

2. Log-In Procedure: Samples are logged in as follows (Refer to Figure 2):

A. Sample Information:

Care should be taken to document the condition of samples upon receipt. Anomalies such as broken or leaking bottles, expired holding times, improper labeling or preservation, and air bubbles in VOA vials should be noted on the chain-of-custody. It should also be noted whether the samples arrived cold.

B. Numbering Samples:

Number the samples on the chain-of-custody form. Record lab numbers used in the black lab book. If no chain-of-custody form accompanied the samples, the sampler must be contacted and one must be filled out. Blank forms are available in the log-in area.

NOTE:
- Sample preparation is required if the samples need to be filtered and/or preserved in the lab.
- If metals are requested, metal digestion is required for all matrices except filtered groundwaters or private well.
- Sample compositing is required if the lab is instructed to composite a series of samples received into one sample for analysis.

C. Subcontracted Work Required:

Refer to the subcontracting SOP for further instructions on subcontracting samples.

D. Special Accounting Notes:

If there is a discount or a special proposal for the sample analyses to be charged, record that information on the chain-of-custody.

E. Labeling Bottles:

1. Record the lab number on the sample bottle label and caps using a water-proof marker.

2. Check that the appropriate preservation and preparation has been circled.

3. If metals and total hardness are both required, using a red water-proof marker, place a dot on the bottle cap of that bottle to be used for those analyses.

F. Preserving Samples:

1. Samples which have been preserved in the field must be checked via pH paper in the laboratory to insure they were preserved to the correct pH. Check preserved samples as follows:

   a. Acid-preserved (HNO_3 or H_2SO_4) samples must be preserved to a pH <2. Using a pH stick, check the pH. If the pH is not <2, note this on the chain-of-custody, then add additional acid until a pH is obtained. IT IS CRITICAL THAT THE CORRECT TYPE OF ACID BE ADDED.

   b. Base-preserved NaOH samples must be preserved to a pH > 12. Using a pH stick, check the pH. If the pH is not > 12, note this on the chain-of-custody, then add additional 10N NaOH until a pH > 12 is obtained.

   c. Base-preserved NaOH with ZnAc samples must be preserved to a pH > 9. Using a pH stick, check the pH. If the pH is not > 9, note this on the chain-of-custody, then add additional 10N NaOH until a pH > 9 is obtained.

2. Samples which have not been preserved in the field need to be preserved in the laboratory. Record any preservation done in the lab on the field sheets or custody records. Refer to Figure 3 for preservation requirements and preserve accordingly. It is important that samples arrive in appropriate containers (see Figure 3). If they do not, contact the inorganic/organic supervisor. They will determine which analyses, if any, can be run.

3. It is important that samples be received and maintained cold for most analyses. If a sample was received warm, make note of this on the field sheet or custody record.

G. Computer Log-In:

Analysis requests are entered into the Laboratory Information Management System (LIMS). The LIMS database is used for scheduling of analytical work and to produce accounting invoices. Analysis requests are entered into the LIMS database as follows:

1. Turn on the computer terminal.

2. Enter: C CHICO (return).

3. Enter: ACSORA (return).

4. Enter: Password (return).

5. At the VMS $ prompt, enter LABMENU (return).

6. Enter: LOGIN (return).

7. Enter: Password (return).

8. The Log-In screen will appear as shown in Figure 2. Using information on the chain of custody, proceed as follows:

   a. Enter project number (return). Account number and name will be filled in automatically. If the information matches the chain of custody, press return.

   b. Sample date automatically defaults to yesterday's date. If this is correct, press return or override with correct date.

   c. Enter sample identification from station location on the chain of custody and press return.

   d. Enter proper matrix number and press return.

   e. Desc: This is used if sample identification is longer than the number of characters in the sample identification field. (Return to override or enter information and return.)

   f. Date received automatically defaults to today's date. If correct, press return or override with correct date.

   g. Days to process automatically defaults to 14 days. If this is correct (standard turnaround time) press return or if rush work, override with the number of days until due.

   h. Duedate: First date listed is report date, second date is the lab due date which can be changed if necessary.

i. Comments: Enter special tests or other information needed to analyze samples correctly and press return.

j. Manager: Filled in automatically; press return if correct or over ride.

k. Price/Cost: Return if correct or over ride.

l. % Markup/Discount: For accounting. Enter a negative number for a discount (i.e. -15 for 15%) and a positive number for markups (i.e. 100 for 2 x price). Check accounting sheets for projects with special pricing.

m. To enter parameters, press **Next Screen** (see Figure 2A).

n. Matrix automatically defaults to matrix entered above (return or override).

o. Enter the proper code name for the test to be run from report MF01. Use parent codes if applicable for quicker entry. (Press the down arrow key, return.)

p. To enter address for the report, press **Next Screen** (see Figure 2B).

q. Type: Report (return).

r. If an address has already been entered and matches the chain-of-custody press **Commit** and return.

s. If no address appears, press return until cursor is in the City field. Type in the city and press return.

t. Type in state (press **Commit** and return). After city and state has been entered for the first sample, it will automatically be copied for the rest of the sample numbers in a single log-in.

9. To duplicate the entry for another sample on the same chain-of-custody:

   a. If next sample to be entered has all the same information as the previous sample entered, press **Duplicate Record** key (F7).

   b. Enter sample identification and press **Commit** key, return.

10. To duplicate only the information in the upper block of the log-in screen (sample date, matrix etc.):

   a. Press **Create Record** key (Insert Here) and enter sample identification.

b. Press **Previous Screen** (F12) if sample date needs to be changed.

c. Press **Next Screen**, return. Then press **Next Screen** again to enter new product codes for the analyses requested.

d. Press **Previous Screen** to enter new sample or to quit.

e. Press **Clear Form** (F17) to enter a new chain-of-custody.

f. Press **Exit/Cancel** (PF4) to exit from the log-in screen.

1. Press **Exit/Cancel** to exit from the LIMS system.
2. At the VMS $ prompt, enter LO (return) to log off the VAX computer.
3. Enter N (return).

13. If in Step 8a. the computer says project number is invalid:

a. Press **Exit/Cancel** (PF4).

b. Enter "Y" (return).

c. On the LIMS Main menu, use the up arrow key to position the cursor on "Seedpak Management". Press **Commit** key.

d. Use the down arrow key to move to project entry. Press **Commit** key.

e. Enter project number (return).

f. Enter account number (9999) and return.

g. Enter description for chain of custody (project name).

h. Enter manager from chain-of-custody (i.e. Finner, K).

i. Press **Exit/Cancel** (PF4), twice.

j. At the LIMS main menu, select log-in and press **Commit**.

14. To print daily log-in reports:

a. Print out the appropriate FS02 reports for only the samples logged in that day. The computer printout must be checked against the custody record or field sheets by the inorganic/organic supervisors so that any errors can be corrected. Print a report as follows:

b. Select "Seedpak Reporting" from the main menu, and press **Commit**.

c. Select FS02 and press Commit.

d. Enter today's date, press Commit and return.

e. The inorganic/organic supervisors are responsible for updating and printing lab schedules as they require them.

f. Any "rush" work is to be brought to the inorganic/organic supervisors' attention immediately.

15. Sample Storage:

a. Proper handling and storage is necessary in order to maintain the integrity of the samples. Refer to Figure 3 for storage requirements.

b. Refrigerated Storage:

1. Store routine custody samples in the walk-in refrigerator. Separate samples by preservative type. Samples with organic analyte requests are stored on a separate shelf in the walk-in.

c. Non-Refrigerated Storage:

1. Metal samples are stored in the log-in area in the appropriate cabinets.

2. Other samples not requiring refrigeration are stored on the appropriate shelves next to the walk-in refrigerator.

d. Any samples removed for analysis must be put back in the original location when the analysis is complete.

16. Sample Handling:

a. Samples are handled in and out of the walk-in coolers and black cabinets by the sample custodian.

b. Analysts are responsible for filling out sample request forms (see Figure 4) to request needed samples.

c. Forms are given to the sample custodian.

d. Sample custodian gathers requested samples and leaves yellow copy of form with samples, and keeps the white copy.

e. Analysts return samples to the walk-in accompanied by the yellow copy of the request form.

f. Next morning, sample custodian puts samples back in their proper place on the shelves.

g. Sample custodian signs off on white sheet that samples were returned. Any remarks are also noted on the white sheet.

17. Storage/Disposal:

Due to limited sample storage facilities available, the following guidelines have been created for sample storage/disposal. Sample storage time may be extended upon client's request.

a. One week after the report issue date, inorganic samples which are in cold storage are to be transferred to warm storage.

b. One month after the report issue date, organic samples which are in cold storage are to be transferred to warm storage.

c. Samples in warm storage should be disposed of as follows:

- Aqueous Samples: Dispose 1 month after report issue date.
- Non-Aqueous Samples: Dispose 6 months after report issue date.

Michael Linskens
Michael J. Linskens
Director, Technical Services

Kim Finner
Kim D. Finner
Laboratory Manager

Revision Date

08/18/87

08/15/88

3-18-90

D2

WARZYN LABORATORY
WASTE LAB PACKAGING

LABORATORY WASTE LAB PACKAGING

Scope and Application: This procedure covers the packaging of samples, waste and expired chemicals generated by the daily activities of the laboratory. All laboratory waste will be packaged by laboratory personnel with the proper Health and Safety training. This procedure is not applicable to groundwater, drinking water or other aqueous samples and waste. For disposal of these materials, see the "Aqueous Waste Pretreatment and Disposal" procedure.

Reference: Lab Pack Procedures, ENSCO

Reagents and Apparatus

1. DOT approved, 17H, 55 gallon metal open top drums with bolt ring sets.
2. DOT approved, 17E, 55 gallon metal drums.
3. Warning Labels.
4. Sample Disposal Labels.
5. Overpack Drums, 85 gallon metal openhead drums with ring-bolt sets.

Notes: Lab Packing can only take place when two or more personnel are involved. Teamwork is required due to safety considerations.

Safety: Staff must wear the appropriate personal protective equipment when handling waste streams.

- Laboratory coat
- Safety glasses
- Gloves
- Respirator
- Splash shield (if needed)
- Tyvek suit

Procedure:

1. **Personnel:** Laboratory personnel will be responsible for organizing, packaging and labelling of laboratory waste. Construction Services will support the laboratory by supplying personnel to help in permitting, securing vendors, transportation and billing. Construction Services will also send personnel to help in the checking and loading of lab packs at the time of shipment.

   Note: All personnel involved with the lab packing are required to have the proper Health and Safety training (40 hours).

2. **Waste stream categories:** The waste that the laboratory generates will be categorized in the following "Waste Streams" for disposal.

   a. Hydrocarbon (H) - The Hydrocarbon stream is composed of solids, oil, soil and sludge samples, except for samples that contain PCBs ≥ 25 ppm.

   b. PCBs (P) - The PCB stream is composed of solids, oil, soil and sludge samples that contain PCBs ≥25 ppm (mg/kg).

c. Organic Solvent (O) - The organic solvent stream is composed of of organic solvent waste generated by daily laboratory processes.

d. Special Waste (S) - The special waste stream is composed of expired chemicals, sample extracts, or wastes that require specialized packaging (i.e., samples that contain large amounts of cyanide, heavy metals or other highly toxic compounds).

Waste Disposal Procedure:: Lab packing will occur quarterly for the above four waste classifications. Lab packing differs for each waste stream and is discussed in the following paragraphs.

Hydrocarbon (H):

1. The Hydrocarbon (H) waste stream will be disposed of in 55 gallon metal open top drums with ring bolt sets (17H). *

2. Soil and solid samples should be disposed of first by removing the jars lid and dumping its contents into the barrel. The jar and lid should then be added to the barrel. It may be necessary to break the jars to make space.

3. Samples that are hard to remove from the jar can be put directly into the barrel and the jar broken in the barrel.

   Note: Caution should be used when glass is being broken. Wear appropriate personal protective equipment.

4. No free flowing liquid should be present in the barrel. Therefore, sludges and oils should only be added when there is enough soil/solid to absorb the liquid. Once the liquid has been added mix it into the drum contents.

5. The barrel should be filled from two-thirds to three-quarters full. Do not over fill the barrels.

6. Analysis may be required before disposal can take place if there is any major change in the waste stream. Contact Construction Services concerning any sampling and testing that may be required.

7. When the barrel is ready for shipment, the ring bolt set can be tightened down and the appropriate labels applied.

   * The barrel should be logged into the waste barrel logbook and the identification number painted on the barrel.

PCBs (P):

1. The PCB (P) stream will be packaged in the same manner as the Hydrocarbon (H) stream except only dispose of waste containing **PCBs ≥ 25 ppm.** Labelled as **PCB WASTE** with a yellow and black PCB label.

2. The drum will be given an identification number from the logbook and that number will be painted on the outside of the barrel.

3. There should be NO free flowing liquid present in the barrel. Oils and sludges should only be mixed with sufficient solids to absorb any free liquid.

4. Fill the barrel until it is two-thirds to three-quarters full. Contact Construction Services concerning any sampling and testing that may be required.

5. Secure the lid with the bolt ring set and apply a second yellow and black PCB label with the correct information.

Organic Solvents (O):

1. The Organic Solvent stream will be packed in 55 gallon metal closed-head drums (17E) for disposal. The drums will be fitted with all brass drum vents to prevent excess pressure from building in the drum.

2. Laboratory generated solvents will be accumulated in 50 L carboys. When the solvent level in the carboy reaches ≈ three-quarters full, the carboy should be taken outside and the solvent should be transferred to the 55 gallon drum by siphoning the solvent out.

3. Water and solids should be kept at a minimum in the Organic Solvent waste.

4. Drums should be filled within 2-3" from the top of the drum.

5. Once the drum has been declared full, the drum vent should be replaced on the drum and the appropriate label(s) attached.

6. It is probable that the company disposing of the waste will require a sample of the solvent before acceptance for disposal. Contact Construction Services concerning any drum sampling that is required.

7. Waste will have to be categorized and packed according to guidelines and requirements of CFR title 49 and any vendor required procedure.

Special Waste (S):

Note: Before any lab packing is initiated, the vendor must be consulted to determine the proper method (i.e., proper container type and size, absorbent, hazard classes, and maximum volume).

1. Place a 4-6" layer of absorbent material (vermiculite) in the bottom of the drum. Be sure the material is evenly distributed.

2. Place a single layer jars, bottles, vials or other of containerized waste on the absorbent. Leave at least 2" of space between each container and the drum side.

3. Cover the waste layer with 2-3" of absorbent.

4. Fill the drum by alternating layers of waste and absorbent.

5. Top off the drum with 4-6" of absorbent to complete the packing.

6. Secure the lid and attach the appropriate labels.

Time Table: Waste will be lab packed quarterly and shipped at least bi-annually. Log-in personnel are responsible for letting the Waste Custodian know when they are running out of room in warm storage. Otherwise, waste samples will be packed quarterly at the Waste Custodian's discretion. Special waste such as expired chemicals will be packed on an as needed basis dictated by the Waste Custodian. The Waste Custodian will be responsible for organizing the lab packing process. It is also the Waste Custodian's responsibility to notify the vendor for pick-up and supervise the loading of the waste.

	Revision Date
Patrick Letterer Patrick M. Letterer Supervisor, Organic Extractables	8-12-91
Gordon L. Gerry Manager, Operations	
Kim Johnson Kim D. Johnson Manager, Analytical Laboratory	

D3

INDUSTRIAL ENVIRONMENTAL ANALYTICAL (IEA)

DO NOT DUPLICATE
COPY

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 The objective of this document is to describe the flow of commercial samples through the Sample Management laboratory.

2.2 Areas to be addressed include a description of each step in the login process and the paperwork involved.

3.0 SAFETY

3.1 Safety glasses, labcoats, and vinyl gloves should always be worn when handling samples.

3.2 Extreme care should be used when handling samples with strong odors or are noted by the client to be hazardous. Such sample containers should be placed in the exhaust hood of the Sample Management area for the duration of the login process.

4.0 APPARATUS AND MATERIALS

4.1 To perform the login process it is necessary to use the following refrigerators: R26, R23, R18, R6, R1, and freezer F1. The Commercial CLP Log is used when such samples arrive at the laboratory. Also required is a SAM computer, a label printer, and a full page printer.

5.0 PROCEDURE

5.1 The process of logging in commercial samples begins at the time they are delivered to the Sample Management laboratory. The first step in this process is to check the top of the box for a white sticker that has the IEA Quote Number on it. If there is a number, call it up on the computer by typing qw and the letter and number from the box (ex. qw

W9109100). Open the box and check the Chain of Custody for a Project Number or a Purchase Order number and see if it matches the Quote. If it does, do a **shift printscreen** to print out the information on the Work Quote Information Screen. Next hit F5 to get to the Analytical screen. Once again, check the Chain of Custody to see if the parameters originally ordered on the quote match what the client has written on the Chain of Custody. If they agree, do a **shift printscreen** here to print out the parameters requested. If a large number of parameters are requested, it will be easier to type qwa space then] (ex **qwa**]). The procedure will generate a list of every parameter the client ordered. This can be accomplished by being on the Work Quote Information Screen, pressing ALT F1, and then typing qwa].

If the quote does not match the Chain of Custody, which will happen, go to the client field on the Quote screen, type in the client's IEA number and hit ALT F5. This gives a list of all of the quotes for this client. Use F3 and F4 to page up and down through the list looking for a match to the Project Number or Purchase Order number from the Chain of Custody. If no match is found, a quote must be created by the login person at the time of login.

When the client's turnaround is known and the parameters requested have been checked we use the five colored stickers to prioritize the projects for login. The colors and their meanings are as follows:

Red - Rush Turnaround
Purple - Limited Protocol (Holding Time)
Green - 5 Day TPH-GC Client
Yellow - 10 Day Turnaround Client
Orange - 15 Day Turnaround Client

The labels are placed on a box flap and can be used in combination to reflect whatever situation the Chain of Custody dictates. Once the label(s) have been placed on the box flap, the date of receipt, method of shipment, and parameters should be written on the flap also as a quick visual reference as to what is in the box.

The sample box(s) are then placed against the wall in order of priority from Rushes and Limited Protocols down to 15 day turnaround clients. Samples having Limited Protocols will be recorded on the Limited Protocol Form (Attachment 5), and the Inorganic department will be notified. At this time the Sample Management person will initial and date each line used and record the name of the person they spoke with.

5.2 Once all of the samples have been prioritized, the next step is to begin setting out the samples on the work table for the login personnel. This is usually done by the Supervisor who, as they are laying out the samples, check for any discrepancies that will require a call to the client for resolution. If there are any discrepancies, a note will be made and kept with the Chain of Custody to alert the login personnel of the problem. The samples should be organized on the table from left to right following the order that they are

written on the Chain of Custody.

5.2.1 When a sample bottle arrives that will require analysis by two or more different departments, the first item to address is if there will be sufficient volume to run all requested parameters. If there is not enough sample to perform all parameters requested, a client discrepancy will be written and forwarded to Client Services to inform the client of the problem.

If there is sufficient sample to run all parameters requested, Sample Management will determine which parameters can be put in the same bottle, i.e. samples with the same preservative or no preservative. The bottles necessary for the split will be taken from the Transpak Area.

Unless Volatile parameters are involved, all splitting of samples will and should be done in the hood in Sample Management. Once the samples are split they should be taken to the Transpak area and preserved (if necessary) to the correct pH required for the tests. Check the preservation table (Attachment 1) for container sizes and pH requirements. A color coded sticker or label will be necessary for any sample that is preserved. See the Preparing Transpak SOP for the appropriate color for each preservative.

If a sample contains both Volatile and non-Volatile parameters, the sample bottle will be stored in the appropriate Volatile refrigerator. INF codes will be used to alert the lab that the Volatiles department needs to analyze its parameters first.

The Notification of Limited Protocol form (Attachment 2) is used when samples arrive to the lab having a limited holding time. The requirements for this form to be used are: less than 8 days remaining on a 14 day protocol, and less than 4 days remaining on a 7 day protocol. Be sure to include the number of samples, matrix, parameter(s) requested, and remaining days until protocol expires when filling out this form. Sample Management fills out the top section of the form and sends the original copy to the Department Supervisor that is affected. The gold copy is sent with the rest of the paperwork to Client Services. The pink copy remains in Sample Management to remind us that we have this discrepancy to resolve as soon as possible.

5.3 The login personnel complete the next step of the process by entering in the information from the Chain of Custody directly into the SAM system.

Begin by typing qw to call up the Work Quote Information screen. Verify the Chain of Custody client information against the screen. Pertinent information that must be filled out includes: Client Representative, P.O. number, and Work ID. If there is a discrepancy in any of this information, use the Chain of Custody as the guide and send up a discrepancy to client services to verify the change. If a quote must be created by Sample Management, it must be forwarded to Client Services for verification at completion of the login process.

Next, press F5 to go to the Work Quote Analytical Screen. Verify that all parameters listed on the Chain of Custody appear on this screen. Parameter codes can be added as needed. To receive the codes, enter the correct number of samples in the "Rec" (received) field and then press F8 and Y. This transfers the Job and Test codes to a Workorder. To go to the newly created workorder, press F5 and chose the "go to ORD" option from the menu.

The number for the project will automatically be chosen and will appear in the "CAT" field. This number should be written in the remarks section of the Chain of Custody. In the "Contact" field, press ALT C to enter "Client Relat". The next field to fill in will be the "Report" field. Enter the appropriate report due date specific to the client. Next, in the "Matrix" field, enter the matrix of the samples (water, soil, oil, etc.). The "Received" field is the next to be filled. Enter the date the samples were received, or it Day 0. The appropriate lab due date should be placed in the "Date Due" field. The "Keep For" field is the number of days IEA will hold the samples after the report is mailed. This is 15 days for standard level I, II, and III QC level projects. Commercial CLP is held for 30 days, unless otherwise specified. EPA work is held for 60 days. The "DISP" field is used to show the method of Disposal. A "D" means normal disposal and the samples were not found to be hazardous. A "R" means the samples for this project will be returned to the client following analysis. A "S" means the samples were found to be hazardous and should be disposed of as such.

The next step involves setting up the sample ID's, parameters, and number of bottles per sample. This is done on the AUTOFRAC screen. To get there, press CTRL F5. Verify the "Samps/Frac" fields are correct at this point. So far, only the "Samps" field will have a number in it. A base sample ID is entered into the "Default ID" field. The "Default Collected Date" should be filled in with the sampling date of the samples. The "Default Category" should be filled in with the matrix of the samples. At this point press F5. This will fill in the Sample ID, Date Collected, and Category fields with the information entered earlier. Here, changes can be made to an individual sample's information. For example, if one sample in ten has a different sampling date, it can be changed in this section.

Next arrow down to the Job and Test codes to verify the correct codes have been entered into the system. When all codes have been entered and/or verified, press ALT F5. This will fill in the "Fracs" field at the top of the screen. The number in the field is the total number of bottles the computer says you have for this receiving event. Make sure this number is correct! Having too few bottles could result in problems later in the process that could result in having to start the data entry process over.

The bottle types were also filled in when ALT F5 was pressed. Page down to the "Location by Fraction Type" field.

Locations for the actual sample bottles are dictated by the parameter being requested. Refrigerator assignments are as follows:

R26: Specific Client Volatiles
R23: Total/Fecal Coliform Samples
R18: Semi-Volatile, Inorganic, TPH, and CLP Samples
R6: GC Volatile Samples
R1: GC/MS Volatile Samples
F1: Charcoal Tubes
Shelfroom: Metals samples, Oils, Paints

Three vials are sent out for GC and GC/MS water volatile samples. All of these vials are placed in a tray in R1 (GC/MS) or R6 (GC).

The Login Checklist (Attachment 4) is the next piece of paper to be filled out. This form is used to make sure all pertinent information has been transferred from the Chain of Custody to the computer system, and the Chain of Custody has been signed and dated.

5.4 After all samples have been created, the next step is to generate a bar code label for the outside of the project folder and labels for each of the bottles in the project. Bar code labels are generated by typing labl_bar and the Workorder Number/f. An example would be **labl_bar 9109100/f.** Bottle labels are generated by typing labl_tst and the Workorder number/f. For example **labl_tst 9109100/f.** Next, the Data For Project sheet (Attachment 3), Data Sheets, and Department Sign Off page must be printed. This is done by returning to a J(1): prompt and first pressing C and enter. This will change the printer selection from the local label printer to the laser printer. Next, type "projmit" and the project number. For example **projmit 149_100.** After all pages have been printed, type A and enter to return the computer to the label printer.

5.5 The Test codes on the SAM generated bottle labels should be matched up with the parameter written on the bottle's label. Be sure to match the Sample ID from the SAM label to the correct sample ID on the bottle. These labels will be subsequently checked by another member of the department to verify the Sample ID's, project numbers, and parameters to be performed. Documentation of this level of review is made on the Login Checklist (Attachment 4) on Line 15. This is one of the most important steps in the login process and extra time should be taken to assure the accuracy of the labels and the bottles they are put on.

5.6 From this point the samples are placed on a cart for the Sample Custodian to distribute to the respective refrigerators. Each lab will then request the samples they need for the day and the sample custodian will deliver them to the lab.

5.7 The last step in the login process is assembling the project folder. The left side of the folder should contain, in the following order: the original Data For Project Sheet, the login checklist, the original Chain of Custody, and then any other paperwork related to the project (ex. Purchase Order, IEA Packing List, Letters, Memos, etc.). The right side of the folder should contain a copy of the Chain of Custody, the SAM Sign Off page and

Data Pages, with any Airbills or Waybills being stapled to the folder. A white sticker should be placed on the folder with the Project Number and Lab Due Date written on it. For Rush projects, a red sticker is used. Five day TPH clients will get a red dot sticker on top of the white sticker. Also, be sure that the bar code label is attached to the front of the folder.

To complete the remaining paperwork, staple a copy of the Chain of Custody to a copy of the Data For Project Sheet, and place them in the box designated for Client Relations Discrepancies.

6.0 SUPPLEMENTAL DOCUMENTS

6.1 SOP for Transpak Preparation, IEA Document Control #SMS00500.NC.
6.2 Login checklist is IEA Document Control Form# SMF00100.NC

7.0 REFERENCES

8.0 SUBSTANTIVE REVISIONS

ATTACHMENT 1

Metals in Water					
Parameter[3]	Technique	Method	Holding Time	Container	Preservation
Aluminum	flame	202.1	6 Months	500 ml P,G	HNO3 to pH <2
	furnace	202.2	6 Months	500 ml P,G	HNO3 to pH <2
	ICP	200.7	6 Months	500 ml P,G	HNO3 to pH <2
	flame	7020	6 Months	500 ml P,G	HNO3 to pH <2
	ICP	6010	6 Months	500 ml P,G	HNO3 to pH <2
Antimony	flame	204.1	6 Months	500 ml P,G	HNO3 to pH <2
	furnace	204.2	6 Months	500 ml P,G	HNO3 to pH <2
	ICP	200.7	6 Months	500 ml P,G	HNO3 to pH <2
	flame	7040	6 Months	500 ml P,G	HNO3 to pH <2
	furnace	7041	6 Months	500 ml P,G	HNO3 to pH <2
	ICP	6010	6 Months	500 ml P,G	HNO3 to pH <2
Arsenic	furnace	206.2	6 Months	500 ml P,G	HNO3 to pH <2
	AA, hydride	206.3	6 Months	500 ml P,G	HNO3 to pH <2
	ICP	200.7	6 Months	500 ml P,G	HNO3 to pH <2
	AA, hydride	7061	6 Months	500 ml P,G	HNO3 to pH <2
	furnace	7060	6 Months	500 ml P,G	HNO3 to pH <2
	ICP	6010	6 Months	500 ml P,G	HNO3 to pH <2
Barium	flame	208.1	6 Months	500 ml P,G	HNO3 to pH <2
	furnace	208.2	6 Months	500 ml P,G	HNO3 to pH <2
	ICP	200.7	6 Months	500 ml P,G	HNO3 to pH <2
	flame	7080	6 Months	500 ml P,G	HNO3 to pH <2
	furnace	7081	6 Months	500 ml P,G	HNO3 to pH <2
	ICP	6010	6 Months	500 ml P,G	HNO3 to pH <2
Beryllium	flame	210.1	6 Months	500 ml P,G	HNO3 to pH <2
	furnace	210.2	6 Months	500 ml P,G	HNO3 to pH <2
	ICP	200.7	6 Months	500 ml P,G	HNO3 to pH <2
	flame	7090	6 Months	500 ml P,G	HNO3 to pH <2
	furnace	7091	6 Months	500 ml P,G	HNO3 to pH <2
	ICP	6010	6 Months	500 ml P,G	HNO3 to pH <2
Boron	colorimetric	212.3	6 Months	500 ml P,G	HNO3 to pH <2
	ICP	6010	6 Months	500 ml P,G	HNO3 to pH <2

Metals in Water-Continued...					
Parameter [3]	Technique	Method	Holding Time	Container	Preservation
Cadmium	flame	213.1	6 Months	500 ml P,G	HNO3 to pH <2
	furnace	213.2	6 Months	500 ml P,G	HNO3 to pH <2
	ICP	200.7	6 Months	500 ml P,G	HNO3 to pH <2
	flame	7130	6 Months	500 ml P,G	HNO3 to pH <2
	furnace	7131	6 Months	500 ml P,G	HNO3 to pH <2
	ICP	6010	6 Months	500 ml P,G	HNO3 to pH <2
Calcium	ICP	200.7	6 Months	500 ml P,G	HNO3 to pH <2
	flame	215.1	6 Months	500 ml P,G	HNO3 to pH <2
	titrimetric	215.2	6 Months	500 ml P,G	HNO3 to pH <2
	flame	7140	6 Months	500 ml P,G	HNO3 to pH <2
	ICP	6010	6 Months	500 ml P,G	HNO3 to pH <2
Chromium	flame	218.1	6 Months	500 ml P,G	HNO3 to pH <2
	furnace	218.2	6 Months	500 ml P,G	HNO3 to pH <2
	ICP	200.7	6 Months	500 ml P,G	HNO3 to pH <2
	flame	7190	6 Months	500 ml P,G	HNO3 to pH <2
	furnace	7191	6 Months	500 ml P,G	HNO3 to pH <2
	ICP	6010	6 Months	500 ml P,G	HNO3 to pH <2
Chromium Hexavalent	Coprecipitation	7195	24 Hours	500 ml P,G	Cool, 4 C.
	colorimetric	7196	24 Hours	500 ml P,G	Cool, 4 C.
	flame	7197	24 Hours	500 ml P,G	Cool, 4 C.
	DPP	7198	24 Hours	500 ml P,G	Cool, 4 C.
Cobalt	flame	219.1	6 Months	500 ml P,G	HNO3 to pH <2
	furnace	219.2	6 Months	500 ml P,G	HNO3 to pH <2
	ICP	200.7	6 Months	500 ml P,G	HNO3 to pH <2
	flame	7200	6 Months	500 ml P,G	HNO3 to pH <2
	furnace	7201	6 Months	500 ml P,G	HNO3 to pH <2
	ICP	6010	6 Months	500 ml P,G	HNO3 to pH <2

Metals in Water-Continued...					
Parameter [3]	Technique	Method	Holding Time	Container	Preservation
Copper	ICP	200.7	6 Months	500 ml P,G	HNO3 to pH<2
	flame	7210	6 Months	500 ml P,G	HNO3 to pH<2
	furnace	7211	6 Months	500 ml P,G	HNO3 to pH<2
	ICP	6010	6 Months	500 ml P,G	HNO3 to pH<2
Iron	flame	236.1	6 Months	500 ml P,G	HNO3 to pH<2
	furnace	236.2	6 Months	500 ml P,G	HNO3 to pH<2
	ICP	200.7	6 Months	500 ml P,G	HNO3 to pH<2
	flame	7380	6 Months	500 ml P,G	HNO3 to pH<2
	furnace	7381	6 Months	500 ml P,G	HNO3 to pH<2
	ICP	6010	6 Months	500 ml P,G	HNO3 to pH<2
Lead	flame	239.1	6 Months	500 ml P,G	HNO3 to pH<2
	furnace	239.2	6 Months	500 ml P,G	HNO3 to pH<2
	ICP	200.7	6 Months	500 ml P,G	HNO3 to pH<2
	flame	7420	6 Months	500 ml P,G	HNO3 to pH<2
	furnace	7421	6 Months	500 ml P,G	HNO3 to pH<2
	ICP	6010	6 Months	500 ml P,G	HNO3 to pH<2
Magnesium	flame	242.1	6 Months	500 ml P,G	HNO3 to pH<2
	furnace	239.2	6 Months	500 ml P,G	HNO3 to pH<2
	flame	7450	6 Months	500 ml P,G	HNO3 to pH<2
	furnace	7421	6 Months	500 ml P,G	HNO3 to pH<2
Manganese	flame	243.1	6 Months	500 ml P,G	HNO3 to pH<2
	furnace	243.2	6 Months	500 ml P,G	HNO3 to pH<2
	ICP	200.7	6 Months	500 ml P,G	HNO3 to pH<2
	flame	7460	6 Months	500 ml P,G	HNO3 to pH<2
	furnace	7461	6 Months	500 ml P,G	HNO3 to pH<2
	ICP	6010	6 Months	500 ml P,G	HNO3 to pH<2
Mercury	cold vapor-manual	245.1	28 Days	500 ml P,G	HNO3 to pH<2
	cold vapor-automated	245.2	28 Days	500 ml P,G	HNO3 to pH<2
	cold vapor-manual	7470	28 Days	500 ml P,G	HNO3 to pH<2

Metals in Water-Continued...					
Parameter [3]	Technique	Method	Holding Time	Container	Preservation
Molybdenum	flame	246.1	6 Months	500 ml P,G	HNO3 to pH <2
	furnace	246.2	6 Months	500 ml P,G	HNO3 to pH <2
	ICP	200.7	6 Months	500 ml P,G	HNO3 to pH <2
	flame	7480	6 Months	500 ml P,G	HNO3 to pH <2
	furnace	7481	6 Months	500 ml P,G	HNO3 to pH <2
	ICP	6010	6 Months	500 ml P,G	HNO3 to pH <2
Nickel	flame	249.1	6 Months	500 ml P,G	HNO3 to pH <2
	furnace	249.2	6 Months	500 ml P,G	HNO3 to pH <2
	ICP	200.7	6 Months	500 ml P,G	HNO3 to pH <2
	flame	7520	6 Months	500 ml P,G	HNO3 to pH <2
	furnace	7521	6 Months	500 ml P,G	HNO3 to pH <2
	ICP	6010	6 Months	500 ml P,G	HNO3 to pH <2
Potassium	flame	258.1	6 Months	500 ml P,G	HNO3 to pH <2
	ICP	200.7	6 Months	500 ml P,G	HNO3 to pH <2
	flame	7610	6 Months	500 ml P,G	HNO3 to pH <2
	ICP	6010	6 Months	500 ml P,G	HNO3 to pH <2
Selenium	ICP	200.7	6 Months	500 ml P,G	HNO3 to pH <2
	furnace	270.2	6 Months	500 ml P,G	HNO3 to pH <2
	AA, hydride	270.3	6 Months	500 ml P,G	HNO3 to pH <2
	ICP	6010	6 Months	500 ml P,G	HNO3 to pH <2
	furnace	7740	6 Months	500 ml P,G	HNO3 to pH <2
	AA, hydride	7741	6 Months	500 ml P,G	HNO3 to pH <2
Silica	ICP	200.7	6 Months	500 ml P,G	HNO3 to pH <2
Silver	flame	272.1	6 Months	500 ml P,G	HNO3 to pH <2
	furnace	272.2	6 Months	500 ml P,G	HNO3 to pH <2
	ICP	200.7	6 Months	500 ml P,G	HNO3 to pH <2
	flame	7760	6 Months	500 ml P,G	HNO3 to pH <2
	furnace	7761	6 Months	500 ml P,G	HNO3 to pH <2
	ICP	6010	6 Months	500 ml P,G	HNO3 to pH <2

Metals in Water-Continued....					
Parameter [3]	Technique	Method	Holding Time	Container	Preservation
Sodium	flame	273.1	6 Months	500 ml P,G	HNO3 to pH<2
	ICP	200.7	6 Months	500 ml P,G	HNO3 to pH<2
	flame	7770	6 Months	500 ml P,G	HNO3 to pH<2
	ICP	6010	6 Months	500 ml P,G	HNO3 to pH<2
Thallium	flame	279.1	6 Months	500 ml P,G	HNO3 to pH<2
	furnace	279.2	6 Months	500 ml P,G	HNO3 to pH<2
	ICP	200.7	6 Months	500 ml P,G	HNO3 to pH<2
	flame	7840	6 Months	500 ml P,G	HNO3 to pH<2
	furnace	7841	6 Months	500 ml P,G	HNO3 to pH<2
	ICP	6010	6 Months	500 ml P,G	HNO3 to pH<2
Tin	flame	282.1	6 Months	500 ml P,G	HNO3 to pH<2
	furnace	282.2	6 Months	500 ml P,G	HNO3 to pH<2
	flame	7870	6 Months	500 ml P,G	HNO3 to pH<2
Titanium	flame	283.1	6 Months	500 ml P,G	HNO3 to pH<2
	furnace	283.2	6 Months	500 ml P,G	HNO3 to pH<2
Vanadium	flame	286.1	6 Months	500 ml P,G	HNO3 to pH<2
	furnace	286.2	6 Months	500 ml P,G	HNO3 to pH<2
	ICP	200.7	6 Months	500 ml P,G	HNO3 to pH<2
	flame	7910	6 Months	500 ml P,G	HNO3 to pH<2
	furnace	7911	6 Months	500 ml P,G	HNO3 to pH<2
	ICP	6010	6 Months	500 ml P,G	HNO3 to pH<2
Zinc	flame	289.1	6 Months	500 ml P,G	HNO3 to pH<2
	furnace	289.2	6 Months	500 ml P,G	HNO3 to pH<2
	ICP	200.7	6 Months	500 ml P,G	HNO3 to pH<2
	flame	7950	6 Months	500 ml P,G	HNO3 to pH<2
	furnace	7951	6 Months	500 ml P,G	HNO3 to pH<2
	ICP	6010	6 Months	500 ml P,G	HNO3 to pH<2

Wet Chemistries in Water					
Parameter [3]	Technique	Method	Holding Time	Container	Preservation
Acidity	titrimetric	305.1	14 Days	100 ml P,G	Cool 4 C.
Alkalinity	titrimetric	310.1	14 Days	100 ml P,G	Cool 4 C.
Biochemical Oxygen Demand (BOD)	5 days, 20 C.	405.1	48 Hours	1000 ml P,G	Cool 4 C.
Bromide	titrimetric	320.1	28 Days	100 ml P,G	none required
Chemical Oxygen Demand (COD)	titrimetric, mid-level	410.1	28 Days	50 ml P,G	Cool 4 C, H2SO4 to pH <2
	titrimetric, low-level	410.2	28 Days	50 ml P,G	Cool 4 C, H2SO4 to pH <2
	titrimetric, high-level	410.3	28 Days	50 ml P,G	Cool 4 C, H2SO4 to pH <2
	automated-colorimetric	410.4	28 Days	50 ml P,G	Cool 4 C, H2SO4 to pH <2
Chloride	colorimetric	325.2	28 Days	50 ml P,G	none required
	colorimetric	9250	28 Days	50 ml P,G	none required
	titrimetric	9252	28 Days	50 ml P,G	none required
	colorimetric	9257	28 Days	50 ml P,G	none required
Cyanide	amenable to chlorine	335.1	14 Days [2]	500 ml P,G	Cool 4 C, NaOH to pH>12 Ascorbic Acid [1]
	spectrophotometric	335.2	14 Days [2]	500 ml P,G	Cool 4 C, NaOH to pH>12 Ascorbic Acid [1]
	Total, UV	335.3	14 Days [2]	500 ml P,G	Cool 4 C, NaOH to pH>12 Ascorbic Acid [1]
	colorimetric	9012	14 Days [2]	500 ml P,G	Cool 4 C, NaOH to pH>12 Ascorbic Acid [1]
Fluoride	distillation	340.1	28 Days	500 ml P,G	none required
	ion selective electrode	340.2	28 Days	500 ml P,G	none required
	colorimetric	340.3	28 Days	500 ml P,G	none required
Hardness, Total	colorimetric	130.1	6 Months	100 ml P,G	HNO3 to pH<2
	titrimetric	130.2	6 Months	100 ml P,G	HNO3 to pH<2
Iodide	titrimetric	345.1	24 Days	100 ml P,G	Cool 4 C.
Methylene Blue Active Substances	colorimetric	425.1	48 Hours	500 ml P,G	Cool 4 C.
Nitrogen Ammonia	colorimetric,phenate	350.1	28 Days	500 ml P,G	Cool 4 C, H2SO4 to pH <2
	distillation	350.2	28 Days	500 ml P,G	Cool 4 C, H2SO4 to pH <2
	ion selective electrode	350.3	28 Days	500 ml P,G	Cool 4 C, H2SO4 to pH <2

Wet Chemistries in Water-Continued...					
Parameter	Technique	Method	Holding Time	Container	Preservation
Nitrogen-TKN	colorimetric,phenate	351.1	28 Days	500 ml P,G	Cool 4 C, H2SO4 to pH <2
	block digestor	351.2	28 Days	500 ml P,G	Cool 4 C, H2SO4 to pH <2
	colorimetric	351.3	28 Days	500 ml P,G	Cool 4 C, H2SO4 to pH <2
	ion selective electrode	351.4	28 Days	500 ml P,G	Cool 4 C, H2SO4 to pH <2
Nitrate	colorimetric, brucine	352.1	48 Hours	100 ml P,G	Cool 4 C.
	colorimetric, brucine	9200	48 Hours	100 ml P,G	Cool 4 C.
Nitrate-Nitrite	colorimetric, hydrazine	353.1	28 Days	100 ml P,G	Cool 4 C, H2SO4 to pH <2
	cadmium reduction, auto	353.2	28 Days	100 ml P,G	Cool 4 C, H2SO4 to pH <2
	cadmium reduction, manual	353.3	28 Days	100 ml P,G	Cool 4 C, H2SO4 to pH <2
Nitrite	spectrophotometric	354.1	48 Hours	100 ml P,G	Cool 4 C.
Oil & Grease, Total	gravimetric	413.1	28 Days	1000 ml G only	Cool 4 C, HCL or H2SO4 to pH <2
	IR	413.2	28 Days	1000 ml G only	Cool 4 C, HCL or H2SO4 to pH <2
	gravimetric	9070	28 Days	1000 ml G only	Cool 4 C, HCL or H2SO4 to pH <2
	gravimetric-sludge	9071	28 Days	1000 ml G only	Cool 4 C, HCL or H2SO4 to pH <2
Petroleum Hydrocarbons	IR	418.1	14 Days	500 ml P,G	Cool 4 C, HCL or H2SO4 to pH <2
pH	electrode	150.1	in-field	50 ml P,G	not applicable
	electrode	9040	in-field	50 ml P,G	not applicable
	test paper	9041	in-field	50 ml P,G	not applicable
Phenolics, T-Recoverable	spectrophotometric	420.1	28 Days	500 ml G only	Cool 4 C, H2SO4 to pH <2
	colorimetric	420.2	28 Days	500 ml G only	Cool 4 C, H2SO4 to pH <2
	4AAP, Manual, Distillation	9065	28 Days	500 ml G only	Cool 4 C, H2SO4 to pH <2
	4AAP, Auto, Distillation	9066	28 Days	500 ml G only	Cool 4 C, H2SO4 to pH <2
	MBTH, Distillation	9067	28 Days	500 ml G only	Cool 4 C, H2SO4 to pH <2
Phosphorus, Ortho	colorimetric, auto	365.1	48 Hours	50 ml P,G	Filter immediately, Cool 4 C
	colorimetric, single	365.2	48 Hours	50 ml P,G	Filter immediately, Cool 4 C
	colorimetric-dual	365.3	48 Hours	50 ml P,G	Filter immediately, Cool 4 C
	total, auto, block digester	365.4	48 Hours	50 ml P,G	Filter immediately, Cool 4 C

Wet Chemistries in Water-Continued...					
Parameter [3]	Technique	Method	Holding Time	Container	Preservation
Phosphorus, Total	colorimetric, auto	365.1	28 Days	50 ml P,G	Cool 4 C, H2SO4 to pH <2
	colorimetric, single	365.2	48 Hours	50 ml P,G	Cool 4 C, H2SO4 to pH <2
	colorimetric-dual	365.3	48 Hours	50 ml P,G	Cool 4 C, H2SO4 to pH <2
	total, auto, block digester	365.4	48 Hours	50 ml P,G	Cool 4 C, H2SO4 to pH <2
Residue (Solids)	filterable (TDS)	160.1	7 Days	100 ml P,G	Cool 4 C.
	non-filterable (TSS)	160.2	7 Days	100 ml P,G	Cool 4 C.
	total (TS)	160.3	7 Days	100 ml P,G	Cool 4 C.
	volatile	160.4	7 Days	100 ml P,G	Cool 4 C.
	settleable	160.5	48 Hours	100 ml P,G	Cool 4 C.
Specific Conductance	meter	120.1	28 Days	100 ml P,G	Cool 4 C.
	meter	9050	28 Days	100 ml P,G	Cool 4 C.
Sulfate	ion chromatography	300.0	28 Days	50 ml P,G	Cool 4 C.
	colorimetric	375.1	28 Days	50 ml P,G	Cool 4 C.
	gravimetric	375.3	28 Days	50 ml P,G	Cool 4 C.
	turbidimetric	375.4	28 Days	50 ml P,G	Cool 4 C.
	colorimetric	9035	28 Days	50 ml P,G	Cool 4 C.
	colorimetric	9036	28 Days	50 ml P,G	Cool 4 C.
	turbidimetric	9038	28 Days	50 ml P,G	Cool 4 C.
Sulfide	titrimetric	376.1	7 Days	500 ml P,G	Cool 4 C, ZnAc/NaOH to pH>9
	colorimetric	376.2	7 Days	500 ml P,G	Cool 4 C, ZnAc/NaOH to pH>9
	colorimetric	9030	7 Days	500 ml P,G	Cool 4 C, ZnAc/NaOH to pH>9
Total Organic Carbon (TOC)	combustion or oxidation	415.1	28 Days	50 ml P,G	Cool 4 C, HCL or H2SO4 to pH <2
	combustion or oxidation	9060	28 Days	50 ml P,G	Cool 4 C, HCL or H2SO4 to pH <2
Total Organic Halides (TOX)	titrimetric	9020	28 Days	1000 ml G only [4] No Headspace	Cool 4 C, H2SO4 to pH <2 [5] Sodium Sulfite
Turbidity	nephelometric	180.1	48 Hours	100 ml P,G	Cool 4 C.

Parameters by Gas Chromatography in Water					
Parameter [3]	Technique	Method	Holding Time	Container	Preservation
Halogenated Volatile Organics	gas chromatography	601	14 Days	3x40 ml vials	Cool 4 C.,Thiosulfate [6]
	gas chromatography	8010	14 Days	3x40 ml vials	Cool 4 C.,Thiosulfate [6]
Non-Halogenated Volatile Organics	gas chromatography	8015	14 Days	3x40 ml vials	Cool 4 C., HCL to pH <2, Thiosulfate [6]
Purgeable Aromatics	gas chromatography	602	7/14 Days [7]	3x40 ml vials	Cool 4 C., HCL to pH <2, Thiosulfate [6]
	gas chromatography	8020	7/14 Days [7]	3x40 ml vials	Cool 4 C., HCL to pH <2, Thiosulfate [6]
Acrolein & Acrylonitrile	gas chromatography	603	14 Days	3x40 ml vials	Cool 4 C., HCL to pH 5, Thiosulfate [6]
	gas chromatography	8030	14 Days	3x40 ml vials	Cool 4 C., HCL to pH 5, Thiosulfate [6]
Phenols	gas chromatography	604	ext.-7 Days anal.-40 Days	1 L, Amber G	Cool 4 C.,Thiosulfate [6]
	gas chromatography	8040	ext.-7 Days anal.-40 Days	1 L, Amber G	Cool 4 C.,Thiosulfate [6]
Phthalate Esters	gas chromatography	606	ext.-7 Days anal.-40 Days	1 L, Amber G	Cool 4 C.,Thiosulfate [6]
	gas chromatography	8060	ext.-7 Days anal.-40 Days	1 L, Amber G	Cool 4 C.,Thiosulfate [6]
Nitrosamines	gas chromatography	607	ext.-7 Days anal.-40 Days	1 L, Amber G	Cool 4 C.,Thiosulfate [6]
Organochlorine Pesticides and PCB's	gas chromatography	608	ext.-7 Days anal.-40 Days	1 L, Amber G	Cool 4 C.,Thiosulfate [6]
	gas chromatography	8080	ext.-7 Days anal.-40 Days	1 L, Amber G	Cool 4 C.,Thiosulfate [6]
Polynuclear Aromatic Hydrocarbons (PNA's)	gas chromatography/LC	610	ext.-7 Days anal.-40 Days	1 L, Amber G	Cool 4 C.,Thiosulfate [6]
	gas chromatography	8100	ext.-7 Days anal.-40 Days	1 L, Amber G	Cool 4 C.,Thiosulfate [6]
	HPLC	8310	ext.-7 Days anal.-40 Days	1 L, Amber G	Cool 4 C.,Thiosulfate [6]

Parameters by Gas Chromatography in Water-Continued...					
Parameter [3]	Technique	Method	Holding Time	Container	Preservation
Haloethers	gas chromatography	611	ext.-7 Days anal.-40 Days	1 L, Amber G	Cool 4 C.,Thiosulfate [6]
Chlorinated Hydrocarbons	gas chromatography	612	ext.-7 Days anal.-40 Days	1 L, Amber G	Cool 4 C.,Thiosulfate [6]
	gas chromatography	8120	ext.-7 Days anal.-40 Days	1 L, Amber G	Cool 4 C.,Thiosulfate [6]
Organophosphorus Pesticides	gas chromatography	8140	ext.-7 Days anal.-40 Days	1 L, Amber G	Cool 4 C.,Thiosulfate [6]
Chlorinated Herbicides	gas chromatography	8150	ext.-7 Days anal.-40 Days	1 L, Amber G	Cool 4 C.,Thiosulfate [6]

Parameters by GC/MS in Water					
Parameter [3]	Technique	Method	Holding Time	Container	Preservation
Purgeables	GC/MS-624 list	624	7/14 Days [7]	3x40 ml vials	Cool 4 C., HCL to pH <2, Thiosulfate [6]
	Priority Pollutant list	624	7/14 Days [7]	3x40 ml vials	Cool 4 C., HCL to pH <2, Thiosulfate [6]
	Hazardous Substance list	624	7/14 Days [7]	3x40 ml vials	Cool 4 C., HCL to pH <2, Thiosulfate [6]
	Target Compound list (TCL)	624	7/14 Days [7]	3x40 ml vials	Cool 4 C., HCL to pH <2, Thiosulfate [6]
	Appendix IX list	624	7/14 Days [7]	3x40 ml vials	Cool 4 C., HCL to pH <2, Thiosulfate [6]
	Priority Pollutant list	8240	7/14 Days [7]	3x40 ml vials	Cool 4 C., HCL to pH <2, Thiosulfate [6]
	Hazardous Substance list	8240	7/14 Days [7]	3x40 ml vials	Cool 4 C., HCL to pH <2, Thiosulfate [6]
	Target Compound list (TCL)	8240	7/14 Days [7]	3x40 ml vials	Cool 4 C., HCL to pH <2, Thiosulfate [6]
	Appendix IX list	8240	7/14 Days [7]	3x40 ml vials	Cool 4 C., HCL to pH <2, Thiosulfate [6]

Parameters by GC/MS in Water					
Parameter [3]	Technique	Method	Holding Time	Container	Preservation
Base-Neutral & Acid Extractables	625 list	625	ext.-7 Days anal.-40 Days	1 L, Amber G	Cool 4 C.,Thiosulfate [6]
	Priority Pollutant list	625	ext.-7 Days anal.-40 Days	1 L, Amber G	Cool 4 C.,Thiosulfate [6]
	Hazardous Substance list	625	ext.-7 Days anal.-40 Days	1 L, Amber G	Cool 4 C.,Thiosulfate [6]
	Target Compound list (TCL)	625	ext.-7 Days anal.-40 Days	1 L, Amber G	Cool 4 C.,Thiosulfate [6]
	Appendix IX list	625	ext.-7 Days anal.-40 Days	1 L, Amber G	Cool 4 C.,Thiosulfate [6]
	Priority Pollutant list	8250	ext.-7 Days anal.-40 Days	1 L, Amber G	Cool 4 C.,Thiosulfate [6]
	Hazardous Substance list	8250	ext.-7 Days anal.-40 Days	1 L, Amber G	Cool 4 C.,Thiosulfate [6]
	Target Compound list (TCL)	8250	ext.-7 Days anal.-40 Days	1 L, Amber G	Cool 4 C.,Thiosulfate [6]
	Appendix IX list	8250	ext.-7 Days anal.-40 Days	1 L, Amber G	Cool 4 C.,Thiosulfate [6]
	Priority Pollutant list	8270	ext.-7 Days anal.-40 Days	1 L, Amber G	Cool 4 C.,Thiosulfate [6]
	Hazardous Substance list	8270	ext.-7 Days anal.-40 Days	1 L, Amber G	Cool 4 C.,Thiosulfate [6]
	Target Compound list (TCL)	8270	ext.-7 Days anal.-40 Days	1 L, Amber G	Cool 4 C.,Thiosulfate [6]
	Appendix IX list	8270	ext.-7 Days anal.-40 Days	1 L, Amber G	Cool 4 C.,Thiosulfate [6]

Metals in Soil					
Parameter [5]	Technique	Method	Holding Time	Container	Preservation
Aluminum	flame	7020	6 Months	100 g P,G	Cool 4 C.
	ICP	6010	6 Months	100 g P,G	Cool 4 C.
Antimony	flame	7040	6 Months	100 g P,G	Cool 4 C.
	furnace	7041	6 Months	100 g P,G	Cool 4 C.
	ICP	6010	6 Months	100 g P,G	Cool 4 C.
Arsenic	ICP	6010	6 Months	100 g P,G	Cool 4 C.
	furnace	7060	6 Months	100 g P,G	Cool 4 C.
	AA, hydride	7061	6 Months	100 g P,G	Cool 4 C.
Barium	flame	7080	6 Months	100 g P,G	Cool 4 C.
	furnace	7081	6 Months	100 g P,G	Cool 4 C.
	ICP	6010	6 Months	100 g P,G	Cool 4 C.
Beryllium	flame	7090	6 Months	100 g P,G	Cool 4 C.
	furnace	7091	6 Months	100 g P,G	Cool 4 C.
	ICP	6010	6 Months	100 g P,G	Cool 4 C.
Boron	ICP	6010	6 Months	100 g P,G	Cool 4 C.
Cadmium	flame	7130	6 Months	100 g P,G	Cool 4 C.
	furnace	7131	6 Months	100 g P,G	Cool 4 C.
	ICP	6010	6 Months	100 g P,G	Cool 4 C.
Calcium	flame	7140	6 Months	100 g P,G	Cool 4 C.
	ICP	6010	6 Months	100 g P,G	Cool 4 C.
Chromium	flame	7190	6 Months	100 g P,G	Cool 4 C.
	furnace	7191	6 Months	100 g P,G	Cool 4 C.
	ICP	6010	6 Months	100 g P,G	Cool 4 C.

Metals in Soil-Continued...					
Parameter [3]	Technique	Method	Holding Time	Container	Preservation
Cobalt	flame	7200	6 Months	100 g P,G	Cool 4 C.
	furnace	7201	6 Months	100 g P,G	Cool 4 C.
	ICP	6010	6 Months	100 g P,G	Cool 4 C.
Copper	flame	7210	6 Months	100 g P,G	Cool 4 C.
	furnace	7211	6 Months	100 g P,G	Cool 4 C.
	ICP	6010	6 Months	100 g P,G	Cool 4 C.
Iron	flame	7380	6 Months	100 g P,G	Cool 4 C.
	furnace	7381	6 Months	100 g P,G	Cool 4 C.
	ICP	6010	6 Months	100 g P,G	Cool 4 C.
Lead	flame	7420	6 Months	100 g P,G	Cool 4 C.
	furnace	7421	6 Months	100 g P,G	Cool 4 C.
	ICP	6010	6 Months	100 g P,G	Cool 4 C.
Magnesium	flame	7450	6 Months	100 g P,G	Cool 4 C.
	ICP	6010	6 Months	100 g P,G	Cool 4 C.
Manganese	flame	7460	6 Months	100 g P,G	Cool 4 C.
	furnace	7461	6 Months	100 g P,G	Cool 4 C.
	ICP	6010	6 Months	100 g P,G	Cool 4 C.
Mercury	cold vapor-manual	7470	28 Days	100 g P,G	Cool 4 C.
	cold vapor-manual	7471	28 Days	100 g P,G	Cool 4 C.
Molybdenum	flame	7480	6 Months	100 g P,G	Cool 4 C.
	furnace	7481	6 Months	100 g P,G	Cool 4 C.
	ICP	6010	6 Months	100 g P,G	Cool 4 C.
Nickel	flame	7520	6 Months	100 g P,G	Cool 4 C.
	furnace	7521	6 Months	100 g P,G	Cool 4 C.
	ICP	6010	6 Months	100 g P,G	Cool 4 C.
Potassium	flame	7610	6 Months	100 g P,G	Cool 4 C.
	ICP	6010	6 Months	100 g P,G	Cool 4 C.
Selenium	ICP	6010	6 Months	100 g P,G	Cool 4 C.
	furnace	7740	6 Months	100 g P,G	Cool 4 C.
	AA, hydride	7741	6 Months	100 g P,G	Cool 4 C.

Metals in Soil-Continued...					
Parameter [3]	Technique	Method	Holding Time	Container	Preservation
Silver	flame	7760	6 Months	100 g P,G	Cool 4 C.
	furnace	7761	6 Months	100 g P,G	Cool 4 C.
	ICP	6010	6 Months	100 g P,G	Cool 4 C.
Sodium	flame	7770	6 Months	100 g P,G	Cool 4 C.
	ICP	6010	6 Months	100 g P,G	Cool 4 C.
Thallium	flame	7840	6 Months	100 g P,G	Cool 4 C.
	furnace	7841	6 Months	100 g P,G	Cool 4 C.
	ICP	6010	6 Months	100 g P,G	Cool 4 C.
Tin	flame	7870	6 Months	100 g P,G	Cool 4 C.
Vanadium	flame	7910	6 Months	100 g P,G	Cool 4 C.
	furnace	7911	6 Months	100 g P,G	Cool 4 C.
	ICP	6010	6 Months	100 g P,G	Cool 4 C.
Zinc	flame	7950	6 Months	100 g P,G	Cool 4 C.
	furnace	7951	6 Months	100 g P,G	Cool 4 C.
	ICP	6010	6 Months	100 g P,G	Cool 4 C.

Wet Chemistries in Soil					
Parameter [3]	Technique	Method	Holding Time	Container	Preservation
Cyanide	spectrophotometric	9010	14 Days	100 g P,G	Cool 4 C.
	colorimetric	9012	14 Days	100 g P,G	Cool 4 C.
Sulfate	colorimetric	9035	28 Days	100 g P,G	Cool 4 C.
	colorimetric	9036	28 Days	100 g P,G	Cool 4 C.
	turbidimetric	9038	28 Days	100 g P,G	Cool 4 C.
Sulfide	colorimetric	9030	-	100 g P,G	Cool 4 C.

Parameters by Gas Chromatography in Soil					
Parameter [3]	Technique	Method	Holding Time	Container	Preservation
Halogenated Volatile Organics	gas chromatography	8010	14 Days	3x40 ml vials[6]	Cool 4 C.
Non-Halogenated Volatile Organics	gas chromatography	8015	14 Days	3x40 ml vials[6]	Cool 4 C.
Purgeable Aromatics	gas chromatography	8020	14 Days	3x40 ml vials[6]	Cool 4 C.
Acrolein & Acrylonitrile	gas chromatography	8030	14 Days	3x40 ml vials[6]	Cool 4 C.
Phenols	gas chromatography	8040	ext.-14 Days anal.-40 Days	100 g ,G	Cool 4 C.
Phthalate Esters	gas chromatography	8060	ext.-14 Days anal.-40 Days	100 g ,G	Cool 4 C.
Nitrosamines	gas chromatography	8070	ext.-14 Days anal.-40 Days	100 g ,G	Cool 4 C.
Organochlorine Pesticides and PCB's	gas chromatography	8080	ext.-14 Days anal.-40 Days	100 g ,G	Cool 4 C.
Polynuclear Aromatic Hydrocarbons (PNA's)	gas chromatography	8100	ext.-14 Days anal.-40 Days	100 g ,G	Cool 4 C.
	HPLC	8310	ext.-14 Days anal.-40 Days	100 g ,G	Cool 4 C.
Chlorinated Hydrocarbons	gas chromatography	8120	ext.-14 Days anal.-40 Days	100 g ,G	Cool 4 C.
Organophosphorus Pesticides	gas chromatography	8140	ext.-14 Days anal.-40 Days	100 g ,G	Cool 4 C.
Chlorinated Herbicides	gas chromatography	8150	ext.-14 Days anal.-40 Days	100 g ,G	Cool 4 C.

Parameters by GC/MS in Soil					
Parameter [3]	Technique	Method	Holding Time	Container	Preservation
Volatile organics	packed column	8240	14 Days	3x40 ml vials[8]	Cool 4 C.
	capillary column	8260	14 Days	3x40 ml vials[8]	Cool 4 C.
Base-Neutral & Acid Extractables	semi-vol packed	8250	ext.-14 Days anal.-40 Days	100 g ,G	Cool 4 C.
	semi-vol capillary	8270	ext.-14 Days anal.-40 Days	100 g ,G	Cool 4 C.

Footnotes

[1] If residual chlorine is present in the sample, 0.6 g of ascorbic acid is utilized. Ascorbic acid is only used if residual chlorine is present.

[2] Maximum holding time is 24 hours when sulfide is present. Optionally, all samples may be tested with lead acetate paper before pH adjustments in order to determine if sulfide is present. If sulfide is present, it can be removed by the addition of cadmium nitrate powder until a negative spot test is obtained. The sample is filtered and then NaOH is added to pH 12.

[3] The following information is based upon EPA requirements as outlined in Table II, Part 136, Title 40 of the Code of Federal Regulations, July 1991. This reference should be consulted if further clarification is desired. Various state agencies have differing requirements for both holding times and preservation from those listed above. In such cases, the local requirements supercede the EPA information.

[4] All samples should be collected in bottles with teflon septa and be protected from light. If this is not possible, use 250 ml bottles fitted with teflon lined caps. Samples should contain no headspace.

[5] If samples contain residual chlorine, it must be removed in the field by adding sulfite to the sample bottle (5 mg sodium sulfite crystals per liter of sample).

[6] If samples contain residual chlorine, 0.008% sodium thiosulfate must be added at the time of sampling and should only be used if residual chlorine is present.

[7] If samples do not receive pH adjustment, the holding time is 7 days. With pH adjustment, the holding time is 14 days.

[8] Alternatively, wide mouth glass jars designed for volatile samples may be utilized with teflon lined caps.

ATTACHMENT 2

NOTIFICATION OF LIMITED PROTOCOL TIME

SAMPLE MANAGEMENT

(D) ORIGINAL: LABORATORY
COPY: 1) CLIENT RELATIONS
COPY: 2) SAMPLE MANAGEMENT

EA SAMPLE NUMBER _____
PARAMETER REQUESTED _____
BY: _____ DATE: _____

DATE RECEIVED _____
SAMPLING DATE _____
REMAINING PROTOCOL _____
TIME: _____

LABORATORY

RECEIVED BY: _____ DATE: _____ TIME: _____

(D) ORIGINAL: CLIENT RELATIONS

ANALYSIS REQUEST:

ACCEPTED	
DECLINED	

NEEDS RUSH	YES		NO	
CALL CLIENT	YES		NO	

SUPERVISOR: _____

CLIENT RELATIONS

CLIENT INFORMED	YES		NO	

(D) ORIGINAL: SAMPLE MANAGEMENT

AUTHORIZATION TO:

CONTACT NAME _____

PROCEED	YES		NO	
INITIATE RUSH	YES		NO	
EXCEED PROTOCOL	YES		NO	

FOLLOW UP COMPLETED BY: _____ DATE: _____ TIME: _____

(D) DISTRIBUTION UPON COMPLETION

* LIMITED PROTOCOL TIME: ≤ 8 DAYS REMAINING OF 14 DAY PROTOCOL TIME.
≤ 4 DAYS REMAINING OF 7 DAY PROTOCOL TIME.

ORIGINAL

ATTACHMENT 3

DATA FOR PROJECT NO. _____

CLIENT	TELEPHONE
CLIENT REPRESENTATIVE	**PROJECT I.D.**
ADDRESS	**PURCHASE ORDER NO.**
	BILLING ADDRESS
	☐ AS LISTED ☐ OTHER (See Below)

Dash #	Client I.D.	Sampling Date	Job Codes	Test Codes	Bottle Size	Location

COMMENTS	TURNAROUND REQUIREMENTS		
		NO. OF WORK DAYS (3)	PRICING FACTOR
	☐ NORMAL	15	1.0 X
	☐ RUSH	_____	_____ X

Date Samples Received	Report Due Date	Lab Due Date	Carrier

TRIX: _____ CUSTOMER SIGNATURE: _____

LAB COMPLETION _____ (Initials) _____ Date FINAL REVIEW _____ (Initials) _____ Date DATE SHIPPED _____ (Initials) _____ Date

1) amples received after 2:00 pm will be assigned the date of the following workday.
2) epresents the date that results are shipped to the customer.
3) Excludes weekends and holidays.

ATTACHMENT 4

SAMPLE LOG-IN CHECKLIST FOR IEA-NORTH CAROLINA

Client _____

1.	Samples were received intact (ie. No Broken bottles, leaks, etc.)	Yes	No
2.	If not, was a notation made on the COC or has a discrepancy form been inititated?	Yes	No
3.	Chain of Custody Form was received? If not, were the parameters and/or client ID's listed on the bottles?	Yes	No
4.	Do the number of bottles received agree with the COC? If not, was any notation made?	Yes	No
5.	Did you sign the Chain of Custody?	Yes	No
6.	Were any transmittal forms or letters sent with the samples?	Yes	No
7.	If a letter was sent, does it match the COC?	Yes	No
8.	Does the Quote match the COC and/or letter?	Yes	No
9.	Does the Quote contain a "Memo"?	Yes	No
10.	If a "Memo" exists, was it printed and placed in the Project folder?	Yes	No
11.	Were shipping documents stapled to the Project folder?	Yes	No
12.	Are there any special requirements to be performed upon receipt (Temp., pH, etc.)? If so, who performed them? _____	Yes	No
13.	Were samples received in a chilled condition? (Cold to the touch).	Yes	No
14.	Was the IEA Project number entered on the COC?	Yes	No
15.	Bottle labels were verified that they correspond with the client's ID's. and that they were placed on the correct containers. Who verified the bottle labelling as correct? _____	Yes	No
16.	Were all INF codes and additional codes logged into the workorder?	Yes	No
17.	Have project Due dates been checked?	Yes	No

The following signature indicates that the individual has performed the above checks during the login process and has brought any discrepancies to the attention of the Sample Management supervisor.

Log-in Completed by: _____ Secondary Review _____

Date of Log-In _____ Date of Sec. Review _____

Time of Log-In _____

ATTACHMENT 5

L TI PF)C

DATE: _____

SAMPLING DATE	BOD	MBAS	O-PO_4	NO_3	TURB	SM	BAC-T	CR^{+6}	CLIENT	LAB NOTIFIED	TIME

SAMPLES WITH LESS THAN THREE PROTOCOL DAYS REMAINING IN PROTOCOL

CLIENT	SAMPLING DATE	PARAMETER	# OF SAMPLES	LAB NOTIFIED	TIME

ACIDITY = 14
ALKALINITY = 14
CYANIDE = 14
TS, TSS & TDS = 7
PHENOL = 7
SULFIDE = 7
REAC. SULFIDE = 7

1.0 APPROVALS

DO NOT DUPLICATE
COPY

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 The objective of this document is to describe the procedure to be followed when subcontracting work to another laboratory.

2.2 Areas to be addressed include paperwork, packaging, and shipment of samples.

3.0 SAFETY

3.1 Safety glasses, labcoats, and vinyl gloves should always be worn when handling samples.

4.0 APPARATUS AND MATERIALS

4.1 All subcontracting work is to be written down on the Subcontracting Log (Attachment 1) at the time of shipment. Transpak coolers will be used to ship all samples to other laboratories.

5.0 PROCEDURE

5.1 The subcontracting process begins in Sample Management with the receipt of the Requisition For Network Order (Attachment 2). These forms will provide Sample Management with all of the information needed to correctly retrieve and ship the samples.

The following information must be provided:

1) IEA Project ID
2) Client Name
3) Sample Numbers to be subcontracted
4) Parameter(s) requested
5) Matrix of samples (Soil, water, extract, etc.)

6) Approval signatures by the QC Director and Lab Director
7) Subcontract laboratory

Take the project ID off of the forms and look up the project using the SAM system to determine the sample's current location and the project's due date. Once the samples have been retrieved from their current location, the next step is to fill out an IEA Chain of Custody form (Attachment 3). See the enclosed attachment for the correct method of completing this form.

5.2 Before the samples are delivered to the Transpak Area for Packaging, make a copy of the Requisition For Network Order and Request To Subcontract Analysis and staple them to a copy of the IEA Chain of Custody. Give the copies to the Department Manager and be sure to place another copy of the Chain of Custody in the Project Folder. The original copies and all samples should then be taken to the Transpak Area for packing and shipment.

5.3 When packaging samples for shipment there are a two rules that must be followed. The first : all volatile samples must be sent in their own Transpak with a trip blank. A trip blank should be included for each method involved. For example, a trip blank for 602 in water would consist of 3x40ml vials of HCL preserved volatile-free water. Trip blanks for 601 in water would consist of 3x40 ml vials of unpreserved volatile-free water. If a combination of unpreserved and preserved methods are ordered, for example 601/602, then only 2 vials per method can be used (2 preserved and 2 unpreserved vials of volatile-free water). When preparing the blanks have a GC/MS operator fill out the instrument ID and data filename on the Blank Request Form (Attachment 4). This information should be written on the Chain of Custody and on the Subcontract Log.

5.4 The Transpak Area will then follow the above rules for packaging subcontracted samples and then prepare the shipment for Federal Express delivery. Any special delivery requirements, like Saturday delivery, should be noted on the Requisition For Network Order.

6.0 SUPPLEMENTAL DOCUMENTS

7.0 REFERENCES

8.0 SUBSTANTIVE REVISIONS

Attachment 1

Week ending _____

IEA

SUB-CONTRACT LOG

DATE SHIPPED	PROJECT NO.	CUSTOMER NAME	SHIPPED TO:

Attachment 2

Requisition For Network Order

trix	IEA Sample ID	Parameter/Method	Detect. Limits	Reptg. Level	Report due date	State Cert.	Unit Price

Workorder No.:_____

. MEMO written? Yes No Please attach if applicable

. Are samples currently in house? Yes No If not, arrival date in house:_____

D. Rush Order? Yes No E. Client contacted? Yes No

. Date protocol holding time expires:_____

G. Samples leached or extracted? Yes No If yes, please attach appropriate paperwork from lab

I. Sample arrival date to network/subcontract lab:_____

. Date results needed from network/subcontract lab:_____

. Did you check for internal execution with:

CT NJ IL FL MA NC VT Other_____ Yes No

(1) If no, "Why?" _____

(2) If "other," is the contractor approved by IEA? Yes No

Requested By Date/Location Lab Manager Authorization Date/Location

Attachment 3

IEA
An Aquarion Company
3000 [illegible]ON
CAR[illegible], [illegible] 27[illegible]
PH # 919-677-0090
FAX # 919-677-0427

CHAIN OF CUSTODY RECORD

NO: 00654

REGULATORY CLASSIFICATION - PLEASE SPECIFY

☐ NPDES ☐ DRINKING WATER ☐ RCRA ☐ OTHER _____

COMPANY

Page _____ of _____

PROJECT #	PROJECT NAME

SAMPLERS: *(SIGNATURE)*

SAMPLE I.D.	DATE	TIME	COMP	GRAB	STATION LOCATION	# OF CONTAINERS	MATRIX SOIL	MATRIX WATER	REQUESTED PARAMETERS					

SUBCONTRACT

REPORT DELIVERABLE REQUEST

- ☐ BLANK DATA
- ☐ DUPLICATE DATA / % RPD
- ☐ SURROGATE / % RECOVERY
- ☐ MS/MSD SUMMARY WITH % RECOVERY, % RPD
- ☐ RETURN SIGNED COC
- ☐ ANALYSTS' FULL NAME
- ☐ DATE ANALYZED
- ☐ DATE EXTRACTED
- ☐ CLP PACKAGE
- ☐ SPECIAL ANALYTICAL REQUEST

RELINQUISHED BY *(SIGNATURE)*	DATE	TIME	RECEIVED BY	DATE	TIME
RELINQUISHED BY *(SIGNATURE)*	DATE	TIME	RECEIVED FOR LAB BY	DATE	TIME

IEA QUOTE NO.	IEA RUSH NO.
PROJECT MANAGER (PLEASE PRINT)	**P.O. NO.**

REMARKS ON SAMPLE RECEIPT

- ☐ BOTTLE INTACT
- ☐ PRESERVED
- ☐ CHILLED
- ☐ CUSTODY SEALS
- ☐ SEALS INTACT
- ☐ SEE REMARKS

IEA REMARKS

FIELD REMARKS

Attachment 4

BLANK REQUEST FORM

NUMBER OF VIALS PRESERVED TIME NEEDED

REQUESTED BY PREPARED BY

INSTRUMENT ID DATA FILENAME

BLANK REQUEST FORM

NUMER OF VIALS PRESERVED TIME NEEDED

REQUESTED BY PREPARED BY

INSTRUMENT ID DATA FILENAME